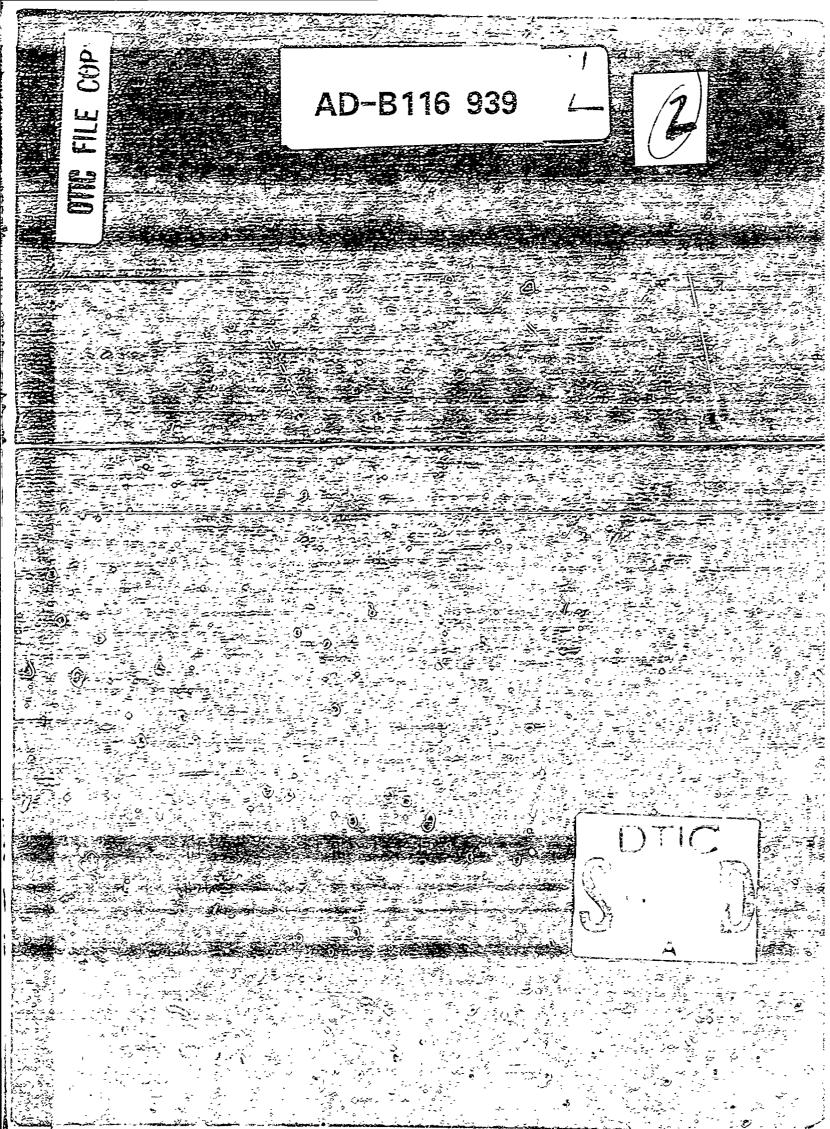
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TITLE
Controlled-Release Personal Use
Arthropod Repellent Formulation - Phase III

TYPE OF REPORT Final Technical Report

AUTHOR Neil A. Randen, Ph.D.

DATE
Typed August 26, 1987
Period of September 21, 1985 through August 31, 1987

Prepared Under Contract Number
DAMD17-85-C-5017 for U.S. Army
Medical Research Acquisition Activity
Fort Detrick, Frederick, Maryland 21701-5014

3M Company
Personal Care Products
St. Paul, Minnesota 55144-1000

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In conducting the research described in the report, the investigators adhered to the "Guide for the Care and Use of Laboratory Animals", prepared by the Committee on Care and Use of Laboratory Animals of The Institute of Laboratory Animal Resources, National Research Council (DHEW Publication No. [NiH] 78-23, Revised 1978).

For the protection of human subjects, the investigators have adhered to policies of applicable Federal Law 45CRF46.

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O.O ABSTRACT

An improved controlled-release arthropod repellent formulation for topical application to a person's exposed skin areas that provides extended protection against biting arthropods, which is safe and agreeable to use, which is more compatible with other current and projected military materials and systems than the Army's current 75% N,N-diethyl m-toluamide (DEET) in alcohol formulation and which complies with the registration requirements of the Environmental Protection Agency (EPA) has been developed in Phase I and Phase II of this contract. The Phase II repellent containing 35% DEET and an acrylate polymer has been refined to improve its low temperature stability. This new Phase III formulation was the basis for the EPA insect repellent registration submitted on May 27, 1987.

The acrylate polymer used in the repellent formulation had to be scaled-up first. A few problems were encountered but they were overcome and the polymer was prepared successfully in the required quantity.) It was shipped to Walgreens.

All the other ingredients for the insect repellent formulation were ordered and sent to Walgreens. A 100 gallon pilot batch was prepared first. The repellent lotion from this batch was very nice. It met all of the product specifications and the lotion was released for packaging. Two thousand eight hundred tubes were filled and sealed on an older, labor-intensive filling machine. The label on the tubes were not centered very well and the seal was not as cosmetically nice as it could have been. These tubes were shipped to 5 different locations for additional field evaluations and testing.

A 1000 gallon production trial batch of repellent lotion was prepared. The manufacturing process seemed to run acceptabily; however, the final cosmetic appearance of the lotion was not quite as good as the pilot batch. The product met its specifications and was released for packaging. A new packaging line was used for the latter and the tubes were very nice. The label was centered and the sealed end of the tube was perfect. These tubes were stored until a disposition can be decided upon.

A technical data package covering the production processes, quality control and product specification was written for the Insect/Arthropod Repellent Lotion.

1.0 INTRODUCTION

Personal Care Products Department of the Consumer Specialties Divsiion of 3M, received the third phase of a U.S. Army Medical Research and Development Command contract to develop a longer lasting, cosmetically acceptable, personal use arthropod repellent formulation. This phase consists of a "demonstration of pre-production pilot manufacturing of the repellent formulation, a demonstration of quality control, a definitization of production processes, finalization of a technical data package, development of a production plan, and registration of the product with the EPA".

Our final Phase II formulation was an oil-in-water emulsioncontaining 35.00 percent N,N-diethyl m-toluamide (DEET) and 5.83 percent acrylate polymer. This formulation provided laboratory 95% protection times against Aedes aegypti mosquitoes of 14-15 hours, 10-11 hours and 14-15 hours in the constant high humidity, variable high humidity and basic hot climatic conditions, respectively. The same formulation evaluated in the field in variable high humidity climatic conditions provided 10.7 + 1.8 hours of complete protection against Aedes sollicitans mosquitoes and 12.3 + 1.8 hours of protection against Anopholes quadrimaculatus mosquitoes. The formulation was shown to be acceptable to 88% of men and women of military age and was shown to be less toxic to animals and humans than the 75% DEET/alcohol formulation. The repellent is packaged in individual 2 ounce. olive drab, high density polyethylene tubes with a noiseless, flip-top cap. The tubes are labeled per the EPA Registration Standard and Guidance Package.

2.0 DISCUSSION

2.1 Phase II Formulation Stability Data

At the end of the Phase II contract, the final candidate formulation was set up in aging studies at 35° F, room temperature and 113° F.

2.1.1 Three Months Data

The evaluation of the Phase II formulation aged at 113°F, room temperature and 35°F was conducted at 3 months. The different lots were checked for viscosity, pH, percent DEET, percent separation and percent weight loss. In addition, freeze/thaw stability data was determined on room temperature samples of each lot. The specific data appear in Figure I,

Appendix A attached. Compared to the initial values, the formulation viscosities of all 3 lots doubled over the 3 months in all the aging conditions. This is not an abnormal occurrence when a Cab-O-Sil thickener is used. For the 113°F samples there was a slight pH drop. Also there was a weight loss for these samples, but it was comparable to what was seen in our Phase II package design studies (See Figure II). The room temperature samples looked normal except for a slight separation evident in some. This separation was more pronounced in the 35°F samples where 6 and 12.3% separations were observed at 1 and 3 months respectively. This low-temperature instability was confirmed when room temperature retain samples were subjected to a freeze/thaw stability tests. A 23% separation occurred on the first cycle.

2.1.2 Six Months Data

The six month stability data appear in Figure III. formulation viscosities and pHs at each condition seemed to have leveled out. They were quite similar to what they were at 3 months (see Figure I). The percent separation of the room temperature and 35°F samples continued to increase. separation also showed itself in the % DEET analysis. The lower values reinforce the fact that DEET had separated from the emulsion phase. There was a 5-6% weight loss from the tubes of the samples aged at 113°F for the 6 months. (Samples aged at 113°F for 3 months are equated to two year of aging at room temperature). The weight loss could be water or water and other ingredients. Based on the percent DEET, which averaged 37.1%, the 113°F aged samples should still be quite effective as an arthropod repellent.

2.2 Improvement of the Phase II Formulation

The separation seen in the $35^{\circ}F$ and the room temperature aging samples of the Phase II formulation was unacceptable. This low temperature instability had to be improved.

2.2.1 <u>Identification of Raw Materials Which Affect Low-Temperature</u> Stability of the Formulation.

The ingredients in the formulation which affected low temperature stability were identified using a freeze/thaw stability test method. Therein, test tubes of the fomulation were frozen for 20 hours and then removed from the freezer. After allowing the tubes to equilibrate to room temperature, they were centrifriged for 15 minutes to accelerate any phase separation. This process was usually repeated for a number of cycles and the percent separation calculated each time.

Room temperature retain samples from the Phase II statistical design experiments were evaluated via the above freeze/thaw method. The following materials were identified as affecting the stability of the formulation. Their effects, positive or negative, are also noted.

Lexol PG-865 (propylene glycol Negative dicaprylate/dicaprate) emollient oil Lexemol AS (glyceryl monostearate) Negative emulsifier Liponic EG-7 (glycereth-7) humectant Negative Carbowax 400 (polyethylene glycol) Positive humectant Varonic LI420 (polyethylene Positive glycol-200-monotallowate) emulsifier Varonic LI48 (polyethylene glycol-Positive 82-glyceryl monotallowate) emulsifier Arlamol E (polypropylene glycol-15-Positive stearyl ether) emollient oil/stabilizer Adol 63 (cetyl stearyl alcohol) waxy Positive emollient/stabilizer Cab-O-Sil M-5 (fumed silica) thickener/ Negative leveling agent Veegum (magnesium aluminum silicate) Positive thickener Natrasol (hydroxyethyl cellulose) Positive thickener.

2.2.2 <u>Statistical Design Experiment to Define Ingredient Effect</u>

The specific effect that each of the above materials had on freeze/thaw stability was determined via a 2^{7-4} fractional factorial design experiment for the first eight ingredients and a 2^3 factorial design experiment for the thickener system. The following regression equations define the effect of each ingredient on the freeze/thaw stability:

Stabilizer Freeze/Thaw % Separation Equation

- % Separation = 13.6% 3.82% (Varonic LI48)
 - 3.6% (Adol 63, Arlamol E)
- + 2.7% (Lexol PG-865) + .9% (Liponic EG-7)
- .8% (Varonic LI420) .4% (Carbowax 400)

Thickener Freeze/Thaw \$ Separation Equation

% Separation = 1.98% + 2.23% (Cab-0-Sil M-5)

- 1.92% Veegum 1.44% Natrasol 1.71% (Cab-O-Sil*Veegum)
 - + 2.67% ($\underline{\text{Cab-0-Sil*Natrasol}}$) + .44% (Veegum*Natrasol) + 4.46% ($\underline{\text{Cab-0-Sil}}$)²

In the equations, the underlined ingredients had a significant effect at \geq 95% confidence interval. A smaller percent separation is desired. Therefore, any ingredient which contributes to reduce the separation will have a negative sign in the equation and those which increase the separation (decrease the stability) will have a positive sign. In other words, more Varonic LI48, Adol 63, Arlamol E, Veegum and Natrasol contribute to a more stable formulation while more Lexol PG-865 and Cab-O-Sil would make the formulation less stable.

2.2.3 Improved Formulations

These regression equations were used to predict which combination of the statistically significant ingredients would provide the most stable formulation with respect to low temperature stability. The formulas are shown in Figure IV. The Phase II formulation in triplicate (75907-11-2, 4, 6) was included for comparison.

2.2.4 Evaluation of New Formulations

The prepared formulations were evaluated for freeze/thaw stability (see Figure V). The Phase II repellent had 4% separation the first cycle and 20% by the fifth cycle. By comparison, the new formulations 462-4-1 through 4 and 75907-11-1, 3 and 5 had essentially no separation through 7 freeze/thaw cycles. The last 3 formulations are replicates of the same formulation. Formulations 462-5-2 and 3 were stable through 5 cycles. Of the freeze/thaw stable formulations, 462-4-1, 2 and 3 and 75907-11-1, 2 and 5 showed 0% separation after 3 weeks at 140° F. Therefore, the choice for a new formulation was between 462-4-1, 2 and 3 and 75907-11-1.

From a toxicological point of view, less of a problem will probably result from a change in the ingredients if they are reduced in concentration as opposed to increasing them. By comparing the new formulas to the Phase II formula, (75907-11-2) in Figure IV, one can see that for 462-4-1, 2 and 3 the concentration of 5 ingredients increase and 3 decrease with respect to the Phase II product. Formulation 75907-11-1 has 3 ingredients decreasing in concentration and only 3 ingredients increasing. Therefore, PCP chose formula 75907-11-1 as the new Phase III formula.

2.2.5 Phase III Versus Phase II Formulation

The new Phase III forumulation was compared to the Phase II repellent to ensure that a loss in aesthetic acceptability and in DEET retention did not result because of the formulation change. Aesthetically the two formulations tested the same with values of 11.1 ± 6.2 and 11.0 ± 5.0 . One would have predicted that the decrease in Cab-0-Sil would have impacted negatively in the aesthetic perception. Apparently the other changes in the formulation offset this.

Evaluation of the six-hour DEET retention for both formulations was also positive. The new formulation retained $74.5 \pm 13.2\%$ DEET on the skin after six hours versus $70.1 \pm 11.0\%$ for the Phase II product.

2.2.6 Toxicological Impact of Phase III Formulation

3M's resident toxicologist was contacted about the changes in the formulation. In his reply (attached - Figure VI) Dr. Griffith stated that the differences between the new Phase III formulation and the Phase II formulation are, toxicologically, insignificant.

2.2.7 Phase III Formulation Laboratory Scale-Up/Sample Preparation

The new formulation was prepared on a larger scale in order to generate enough material for additional longer-term stability studies. Samples of this formulation were set to Dr. Raj Gupta (LAIR) (12) and to Colonel Reinert at Fort Detrick (36). The samples were going to be used for mosquito repellency evaluations to ensure that the formulation change did not affect its efficacy.

Larger three thousand gram batches of the new Phase III lotion were also prepared to demonstrate that the formulation could be scale-up successfully. Colonel Reinert requested 100-150 tubes of this repellent to be used for additional evaluations. Therefore, 100 tubes were packaged and sent.

2.3 Environmental Protection Agency

To be able to ship the repelient lotion manufactured at Walgreens for Phase III of this contract, the product had to be registered with the Envionmental Protection Agency (EPA). The registration process at EPA can take up to 180 days or 6 months; therefore, the data package for the new Phase III repellent lotion had to be finalized and sent in as soon as possible if PCP had any intentions of trying to ship the final product by the end of the contract.

2.3.1 Registration of the Insect/Arthropod Repellent Lotion.

The bulk of the registration package for the EPA had been put together during Phase II of the contract. However, the low temperature instability problems of the Phase II formulation which surfaced after the completion of Phase II necessitated a reformulation to obtain a new, more stable Phase III formulation. These changes were incorporated into the EPA registration package.

Dr. Arturo Castillo, Product Manager of the Insecticide-Rodenticide Branch of the EPA was contacted about Personal Care Products' concerns on the formulation change and whether PCP would have to do any toxicity retesting. He stated that if the changes were small and if they had little toxicological impact on the formulation, then PCP could use their Phase II toxicity data. He also stated that the EPA would make the final decision whether or not the changes had a toxicological impact. In addition, he informed PCP of a new PR Notice 86-5 that the data package had to follow. Because of the urgency of our application he also suggested that we hand deliver the

application to the EPA. Then if any changes needed to be made, they could be taken care of right there. The registration data package was delivered to Mr. Tavano of the EPA on May 27, 1987 by Dr. Frank Griffith (3M Toxicolocy) and the author. It was reviewed by Mr. J. Tavano to make sure it was complete and by Mr. J. Carlee's group to ensure that the format was correct. One additional form was needed which was filled out in Mr. Tavano's office.

A receipt acknowledging PCP's request for registration of an "Insect/Arthropod Repellent Lotion" and a "Report of Analysis for Compliance with PR Notice 86-5" were received from the EPA. In the latter PCP was informed that Good Laboratory Practices (GLP) approvals prior to the toxicological studies were not sufficient. Therefore, the proper forms, signed after the fact that GLPs were followed, were obtained and forwarded to the EPA. In addition, the legibility of some of the material in a couple of studies was reported as "marginal". They were referring to a copied gas-chromatographic DEET analysis of the repellent lotion which did not reproduce very well. This assay was rerun and better copies were sent to Mr. Castillo. A cover letter listing the corrections is attached in Appendix A, Figure VII.

A copy of the registration package was sent to Colonel Reinert and to Mr. L. Rutledge (LAIR). Also, a copy of PR Notice 86-5 was sent to Colonel Reinert.

2.3.2 Shipment of 2,000 Samples of Production Repellent Lotion

Personal Care Products/3M had been requested to send 2,000 2-ounce tubes of repellent to the Army for evaluation prior to the probable issuance of a final EPA registration number for said product. A letter was put together explaining the situation to the EPA (copy sent to Colonel Reinert). Personal Care Products/3M's position was that this shipment of product does not go against the intent of the law. A company can manufacture a pesticide product at a plant and ship it back to the parent company for evaluation. In this instance, PCP/3M was working for the Army, under a contract, and would simply be shipping a "sample" of the manufactured product back to the parent company, the Army. The Army was going to use the material for research purposes for additional, controlled evaluations.

The letter was delivered to the EPA on May 27, 1987. No response was received by the last week in June so Mr. Tavano (EPA) was contacted by phone. Mr. Craig Sterling also contacted Mr. Tavano and a number of other people before

permission was finally obtained to ship 2,000 samples. No special labeling was required on the shipping boxes. A letter was enclosed with each box stating that the samples were for research testing only.

2.3.3 Shipment of 38,000 Tubes of Repellent

The Phase III contract calls for the delivery of approximately 38,000 additional tubes of repellent lotion to the Army. To ship these tubes a number of criteria will have to be met: an EPA registration number will have to have been issued; this number probably will have to appear on the package; and the label on the tube will have to comply with all of the EPA requirements and have their approval. If all that had to be changed was the EPA number on the tubes, a pressure sensitive label containing the appropriate numbers could be applied. Of course, this would mean re-handling the tubes which would involve some additional expense. Personal Care Products/3M will have to wait and see what will be required and if it is still desired that the tubes be shipped.

2.4 Acrylate Polymer Manufacture

The experimental polymer used in the repellent lotion had been prepared in small quantities in the laboratory during Phase I and Phase II of this project. The polymer required for the Insect/Arthropod Repellent Lotion production at Walgreens dictated that the polymer be prepared on a larger scale also. Therefore, the polymer was prepared at Chemolite (a 3M manufacturing plant) during the first week of June. The polymerizations were scheduled to be run in 1000 lb. batches.

As with any new manufacturing procedure, the first run or two constitute a learning process for the production personnel, as well as a time to make adaptations in the procedure to make a better product. Such was the case here; the first production batch had to be scrapped. In the second run an exotherm occurred and polymer with a 3300 cps. viscosity was obtained. This was a little below the visiosity specification; so the polymer was set aside for future disposition. The third production run went very well. The reaction exothermed in a very controllable manner and was complete after a conversion of monomer to polymer of greater than 99.5% was obtained. The final polymer diluted in the laboratory to 25% polymer in DEET gave a Brookfield viscosity of 5550 cps. The target was 6000 cps. with an acceptable range of 4500 - 7500 cps. Unfortunately, the lot was mistakingly diluted to 21.66% polymer because of problems associated with an in-process test.

However, as long as the percent polymer was known this was not a problem since the polymer was already shown to be within specifications.

Approximately 2000 lbs. of the polymer was shipped to Walgreens in Chicago for the compounding in the "Insect/Arthroped Repellent Lotion."

2.5 Repellent Lotion Manufacture

A manufacturing process for the production of the Insect/Arthropod Repellent Lotion based on laboratory experiences appeared in the Phase II Technical Data Package. This process in turn was adapted to production equipment by Dr. P.K. Sundaram (at Walgreens). The major concern in scaling-up a process such as this, was whether or not the different kettles, mixers, homogenizers, etc. used in the production would drastically affect the final product. That is, would the production lotion be as good as the laboratory lotion? Hopefully, it would be better.

2.5.1 Pilot Batch (100 Gallons)

The initial production of the repellent lotion was scheduled to take place in a 100 gallon kettle. This size was chosen because it was intermediate between the 3 gallon laboratory batches and the 1000 gallon production trial batch to be run later. If any problems in the process were encountered, the appropriate changes could be made in the manufacturing process prior to the 1000 gallon batch. In addition, if the product turned out to be unsatisfactory then a smaller amount of material would have to be disposed and then another pilot batch could be prepared.

The 100 gallon pilot batch was run on July 6, 1987 at Walgreens in Chicago. The manufacturing process went very smoothly. The reverse blade agitation in the kettle gave excellent mixing and as a result the final Insect/Arthropod Repellent Lotion looked very nice. A yield of 102% of homogenized lotion was reported (see attached Appendix B Pilot Batch manufacturing records). The lotion had an average DEET content of 34.7% and a lotion viscosity 163,000 (#TE @ 0.6 rpm) (21,600 cps - # TB @ 5 rpm). The specifications are 31.58 to 36.75 percent DEET and 150,000 -- 250,000 cps respectively. The viscosity of the lotion will probably increase some with time. A copy of the certificate of analysis for the lotion is attached at the end of the Pilot Batch manufacturing process in Appendix B. The lotion was released for packaging.

2.5.2 Production Trial Batch (1000 Gallon)

The minor manufacturing process improvements suggested in the pilot batch were incorporated into the process for the 1000 gallon production trial batch. Again, a different kettle and different stirrers, etc. would be used for this larger batch as compared to the pilot batch. In addition, it would take more time to heat this larger batch up to temperature and conversely more time to cool the batch. Would these as well as the other changes affect the final product?

The production trial batch was run at Walgreens on July 27, 1987. The preparation of the water and the oil phases for the formulation went very smoothly. However, a problem encountered when the water phase was added to the oil phase. Usually this addition was done with the agititators (stirrers) turned up very high, to get efficient mixing. This was what the manufacturing process specifications requested. The equipment configuration dictated that the stirrers be turned off during the addition of the water phase to the oil phase. When the addition was finished, they were turned back on and the process completed. At the end, just prior to homogenization, the final formulation did not appear to be as creamy as the pilot batch. For this reason the pressure on the sonatator was increased from 400 to 800 psi and a smaller orifice was used in an attempt to improve the appearance of the formulation. These changes did help the production trial batch lotion but it still was not as nice as the pilot batch. The final lotion did meet all of the release specifications with 35.3% DEET content and a viscosity of 222,300 cps.

A copy of the certificate of analysis is attached of the production process (Appendix B). The yield for the manufacturing process again was 100%.

The production trial batch was released for packaging.

2.5.3 Manufacturing Clean-Up

The repellent lotion is a tenacious product by design when placed on the skin. It is also a hard product to clean from equipment. Walgreens experienced a clean-up problem for both the pilot and the production trial batches.

A procedure to clean the production equipment was developed in the lab. It consisted of heating an aqueous 4% solution of "Alconox" (Alconox, Inc.; New York, NY 10003) to 80° C for 30 minutes. The kettle was then rinsed with hot water to remove the soap film.

2.5.4 Accelerated Stability Testing of the Manufactured Lotions

Accelerated low-temperature stability of the pilot batch and the production trial batch was determined by subjecting samples of the formulations to freeze/thaw cycling with centrifuging between to accentuate any separation. The data is in Figure VIII.

One can see that the pilot batch had freeze/thaw stability (0% separation) very similar, actually better, than the laboratory prepared Phase III formulation. By comparison the production trial batch formulation was not quite as stable with a separation of 3-4 percent. The encouraging thing was that the separation of the latter did not increase as additional freeze/thaw cycles were run. In addition the latter was definitely more stable than the Phase II formulation.

The pilot batch formulation was as stable as any laboratory prepared product. The production trial batch formulation was not quite as stable as hoped, but more so than the previous Phase II formulation.

2.5.5 Production Summary and Proposed Recommendations

The pilot production batch of the Insect/Arthropod Repellent Lotion went very well at Walgreens. The final product looked very nice and met Personal Care Products/3M's freeze/thaw stability criteria. This demonstrated that the lotion could be prepared on a larger production scale satisfactorily.

The larger scale production trial batch appeared to go nicely also. However, the lotion from this batch, while meeting all the specifications etc. was not quite as nice as the pilot batch in its appearance and in its freeze/thaw stability. These differences are probably do to the fact that the kettle agitation was turned off when the water phase was being added to the oil phase. This was contrary to the written procedure in the manufacturing specifications and contrary to the acceptable procedure for making a good, stable emulsion.

Personal Care Products/3M recommends that the 1000 gallon manufacturing process be repeated as a second production trial batch prior to letting this product out for procurement for future Army needs. In a situation like this, where it is realitvely certain that good product will be produced, Personal Care Products/3M would normally have the product packaged. Then the material would be sold if it was acceptable.

A more economical approach to demonstrate that acceptable material can be prepared in the 1000 gallon kettle would be simply to manufacture 800 gallons of the lotion and then store it in 55 gallon drums.

This bulk stored lotion could be packaged into tubes with EPA approved labels at a later date.

A cost proposal has been put together covering a second production trial at Walgreens in which the lotion would be bulk packaged. It is attached in Appendix D.

2.5.6 Final Manufacturing Process Specifications

All of the recommended changes suggested by the pilot batch and the production trial batch have been incorporated into a final manufacturing process. It is attached in Appendix B.

2.6 Packaging

The 2 ounce, olive-drab, high density polyethylene (HDPE) tube was the preferred package for the insect/arthropod repellent lotion at the end of the Phase II contract.

2.6.1 Dispensibility

During the interm between Phase II and Phase III the percent dispensibility from the HDPE tube was questioned. It was felt that a higher percent of the formulation should be dispensed. Additional studies were conducted comparing both the Phase I and Phase II formulations in HDPE tubes and LDPE (low density polyehylene) tubes. From the data in Figure IX it can be seen that a slightly better dispensibility was obtained from the more pliable LDPE (92.1%) than from the HDPE (88.9%) tubes. However, dispensibility is not the entire story for tube construction. When the long term aging data of the lotion in both tubes are compared it can be seen that there was a lower weight loss from HDPE tubes (1.8%) than from the LDPE tubes 5.2%. (See Figure II).

Based on these comparisions the HDPE tube was still the tube construction of choice for Phase III.

2.6.2 Label and Tubes

The U.S. Army requested 2000 units of packaged product from a manufacturing run be shipped to them for evaluation in the field. The product was to be received by July 1, 1987.

Obtaining the tubes necessary for the production lotion took a little longer than anticipated. The problem appeared to be the number of times that the label copy for the silk-screen printing had to be sent back and forth between 3M and the tube manufacturer for corrections. This caused a delay in tube manufacture and necessitated a contract modification on the shipment of the 2000 samples to the Army from July 1 to July 13, 1987.

After the above there are still corrections that need to be made on the label. The word (cream) following "Insect/Arthropod Repellent Lotion" on the front label was suppose to be removed. The Skin Application "directions" were suppose to be in larger print. There is ample room on the back of the tube to do the latter. Also, actual numbers will have to replace the XX's for the following items:

6840-00-XXXXX
TYPE (XXX)
Federal Specification XXXXX
EPA Reg. No. XX
EPA Est. No. XXXX

In addition the EPA is reviewing the "draft label" to ensure that it meets all of their criteria. If they require any changes, these also will have to be made to the "final Label."

2.6.3 Pilot Batch

The 2000 requested samples for the Army were obtained from the 100 gallon pilot batch. They were filled on an older (KALEX) filling machine. The cosmetic appearance of the final tube seal was not as good as it could be. It was wavey in appearance and puckered the tube near the seal. In addition the front and back labels weren't centered very well. Personal Care Products was informed that these faults would be corrected on the 1000 gallon production trial batch.

The filled tubes were sent to the following addresses:

800 tubes

President
U.S. Army Armor Engineer Board
ATTN: ATZK-AE-EN (Capt. Lee)
Fort Knox, KY 40121-5470

400 tubes

Mr. Karl Schreck

Insects Effecting Man and Animals Research

Laboratories

P. O. Box 14565, USDA Gainesville, FL 32604

400 tubes

Commander

Letterman Army Institute of Research

Dept. of Cutaneous Hazards

ATTN: SGRD-UL-CH (Mr. Louis C. Rutledge)

Presidio of San Francisco, CA 94129

100 tubes

Product Manager for Arthropod Repellents U.S. Army Medical Material Development

Activity

ATTN: SGRD-UMB (Col. Reinert)

Bldg. T-622 Fort Detrick

Frederick, MD 21701-5009

300 tubes

3 M

2.6.4 Production Trial Batch

The packaging of this product on a new filling machine proceeded very smoothly. The labels on these tubes were centered properly and the tube seal was very nice. The only problem encountered on a few of the tubes was a V in the sealed end . This was caused by the tubes being slightly too short. Therefore the specifications for the tube will be increase 1/4" in length to 3 13/16". These filled tubes will be stored until a suitable disposition can be decided upon.

The packaging specifications are attached in Appendix C.

2.7 <u>Technical Data Package</u>

A Phase III Technical Data Package was put together for the manufacture of the Insect/Arthropod Repellent Lotion. It included the manufacturing process, quality control and specifications for the manufacture of the repellent lotion.

2.8 Production Plan

A production plan which contains all the time-phased production actions will be sent separately.

2.9 <u>Premanufacturing Notice</u>

A premanufacturing notice (PMN) for the EPA will have to be put together on the acrylate polymer in DEET before this ingredient can be used for a non-research and development use. Use in a commercial product would constitute such a use.

The process of putting together a PMN will take up to "six (6) months" to finalize. This time frame has to be taken into consideration when any 3M "Insect/Arthropod Repellent Lotion" is ordered in the future.

2.10 Extended Shelf Life Studies

The Phase III contract was extended in response to a request from the U.S. Army Medical Research Acquisition Agency to modify 3M's proposal for extended shelf life studies of the Phase III production formulation dated January 23, 1987. It was specifically requested that all testing proposed for the three month and the six month time intervals be removed and then added as an extension to the current Phase III contract. The proposed extended shelf life study would include only the studies conducted at one, two, three, four and five year intervals. Figure X lists the stability tests, aging conditions and time frames for the studies.

2.11 User Training Package

The detailed training package developed during Phase II for procedures, techniques, amounts of repellent formulations to apply, safety considerations, etc. for training the user was refined after the "Draft Final Report" of Phase II and the changes were included in the Final Technical Report for Phase II.

3.0 SUMMARY

A low temperature instability problem surfaced in the aging studies of the final Phase II Insect/Arthropod Repellent Lotion. Those ingredients which contributed positively or negatively to the formulation stability were identified via freeze/thaw studies of retain samples. Once identified the relative effect of each was determined via statistical design experiments. In this manner an improved Phase III formulation was defined which was far superior in its low temperature stability. The changes in the new formulation were kept to a minimum in order to have as small of a toxicological impact as possible.

The Phase III formulation was as aesthetically pleasing as the Phase II formulation. In addition the new formulation retained slightly more DEET on the skin via the 6-hour DEET substantivity test.

The Phase II EPA data package was changed to reflect the differences in the new Phase III for mulation and to conform to the PR Notice 86-5. Dr. Frank Griffith (3M Toxicologist) stated that the differences were small and were toxicologically insignificant. The EPA will assess the changes and make the final decision of whether or not PCP/3M will have to do any additional toxicity testing. The data package was submitted to the EPA on May 27, 1987.

The acrylate polymer used in the Insect/Arthropod Repellent Lotion also had to be scaled-up from laboratory size to production. This was carried out at Che molite. facility manufacturing with in early June. As most manufacturing process scale-ups, a learning curve is usually encountered and this turned out to be the case here. By the third production run most of the problems had been worked out and very good polymer was finally prepared. The only glich occurred with a new in-process test to measure the percent polymer in the solution. It gave erroneous results causing the polymer to be diluted to 21.67%. However, as long as the percent polymer in the solution was known this was not a problem.

The acrylate polymer as well as the other ingredients required to prepare the repellent lotion were received by Walgreens in Chicago. Difficulties in obtaining the labeled tubes caused the production to be postponed until the 6th of July. The first pilot batch of the lotion was 100 gallons. The formulating process went very nicely. The final product met all of its specifications and was released. Two thousand units of this product in 2 ounce HDPE tubes were packaged on an older filling line. The labels were not lined up on the tubes as accurately as they should have been nor were the tube seals as cosmetically acceptable as they could have been. The yield on the formulation process was recorded as 102%. A realistic packaging yield was not obtained because only half of the lotion could be packaged because of the limited number of tubes on hand.

The 2000 samples were sent to five different locations for additional field evaluations and other tests.

After the initial pilot batch, a 1000 gallon production trial batch was prepared. The process went okay; however the final lotion did not look as good as the pilot batch lotion. Later it was learned that the agitation in the kettle had been turned off during the combination of the water and oil phases. This may explain why the formulation was not as smooth as the pilot batch. As before the yield for the production trial batch lotion was 100%. The lotion met all of its specifications. It was packaged on a new IWKA filling line into the 2 ounce HDPE tubes. The seal on the tubes was very nice this time and the labels were aligned perfectly. The packaging yield was 94.0% acceptable tubes, 4.5% destroyed tubes and 1.5% of the tubes unaccounted for.

Freeze/thaw stability studies were run on the production repellent lotions. The pilot batch lotion was as stable as any laboratory prepared lotion, while the production trial batch lotion had 2-4% separation after 7 cycles. The old Phase II formulation had 20% separation by the 5th cycle.

In addition, a technical data package was written covering the production, quality control and specification for the manufacture of the repellent lotion.

4.0 CONCLUSION

The EPA registration data package for the new Phase III Insect/Arthropod Repellent Lotion has been delivered. A Technical Data Package for the production of the repellent lotion has been written as well as a Production Plan.

The scale-up of the acrylate polymer, used in the lotion, went quite nicely after the inital runs. As with any polymerization it has to be monitored quite closely during the key reaction time period. Very few problems should be encountered with this process in the future.

Two production batches of the Repellent Lotion were prepared by Walgreens. In the pilot the product obtained was very nice; however, it was packaged on an older piece of equipment and the cosmetic appearance of the tubes was not as good as it could have been. In the production trial a miscommunication resulted in repellent lotion which wasn't quite as good as the pilot batch. The lotion met all of its release specifications and was packaged on a new filling line. The finished tubes were very nice.

It has been demonstrated that the repellent lotion can be prepared and packaged satisfactorily on a production scale. What remains to be demonstrated is that these two functions can be accomplished for the same production run. I don't see this as a serious problem. I believe that good repellent lotion can be prepared and packaged on a production scale.

5.1 Appendix A - Figures

FIGURE I

PHASE II REPELLENT FORMULATION STABILITY

	<u>Initial</u>	Viscosity (cps)	рН	% DEET	% SEPARATION	% WEIGHT LOSS
	49-47-4 49-52-1 49-52-2	153,000 134,000 122,000	7.15 7.12 7.22	(38.2) (38.2) (38.2)	0 0 0	Q 0 0
	Room Temperature	<u>.</u>		•		
	49-47-4 49-51-1 49-51-2	373,000 315,000 311,000	7.06 6.88 6.93	39.9 38.6 40.0	2.0 0.0 2.0	.2 .2 .2
	113°F - One Mont	<u>.h</u>				
	49-47-4 49-51-1 49-51-2	511,000 589,000 661,000	7.26 7.23 7.33	38.3 38.5 38.5	0 0 0	.5 .7 .7
(.	113°F - 3 Months	<u>.</u>		•		•
	49-47-4 49-51-1 49-51-2	357,000 415,000 304,000	6.86 6.82 6.77	42.0 38.5 41.2	0 0 0	2.1 2.0 2.0
	Freeze/Thaw					
	49-47-4 49-52-1 49-52-2		7.21 7.13 7.20		23.3 23.3 23.3	0 0 0
	35°F - One Month	<u> </u>		,		
	49-47-4 49-52-1 49-52-2	425,000 300,000 410,000	7.33 7.23 7.34	39.9 37.4 36.8	2.0 7.7 9.0	0 0 0
	35°F - 3 Months		·			
C	49-47-4 49-52-1 49-52-2		der 140 für 150 jur 150 für 150 150 für 150 str.	****	12.5 12.0 12.5	0 0 0
~~~						

FIGURE II*

#### PACKAGE AGING DATA

PACKAGE COMPOSITION	PERCENT WEIGHT LOSS ROOM TEMPERATURE	(2 MONTHS) 120(o)F
LDPE 1004 overcoat	•4 % ·	7.6%
LDPE Phase I tube 1004 overcoat	.3%	5.6%
LDPE UV; TP-46	.2 %	5.2%
HDPE UV; TP-46	.1%	1.8%

LDPE = low density polyethylene HDPE = high density polyethylene

^{*} Figure XXX from Phase II Final Technical Report

FIGURE III*

PHASE II REPELLENT FORMULATION - SIX MONTH STABILITY

% WEIGHT LOSS	000	4.0 0.0 5.0	0 0 5 5 8	0.0 0.1 0.0
% SEPARATION	000	⊢00	8.3 ³ 0 6.7 ³	203 143 18 ³
% DEET	$\binom{38.2}{38.2}$	37.0 37.1 37.3	33.4 ² 35.8 34.4 ²	31.62 31.52 30.72
됩	7.15 7.12 7.12	6.97 6.69 6.70	7.15 7.17 7.16	7.19 7.25 7.28
VISCOSITY (cps)	153,000 134,000 122,000	530,000 424,000 486,000	376,700 332,800 314,600	396,400 438,100 488,800
FORMULATION	49-47-4 49-52-1 49-52-2	49-47-4 49-52-1 49-52-2	49-47-4 49-51-1 49-52-2	49-47-4 49-51-1 49-51-2
CONDITION	Initial Values	113°F	Room Temperature	35°F

Average value at one month
 Formulation separation caused less DEET to be in emulsified portion
 Approximate values because of difficulty in reading

^{*} Figure VI, May 1987 monthly report, Phase III.

FIGURE IV** - NEW FORMULATIONS

Formulation				INGRED	IENT AMOUN	INGREDIENT AMOUNT - PERCENT		
Number	Cab-0-Sil	Natrasol	Veegum	Arlamol E	Adol 63	LexolPG865	Lexemul AS	Varonic L148
462-4-1	1.64	.575	.81	2.20	2.20	2,41	1.55	1.65
462-4-2	1.65	• 65	.83	2.20	2.20	2.41	1.55	1.65
462-4-3	1.70	•80	.74	2.20	2.20	2.41	1.55	1.65
462-4-4	1.73	.80	.71	2.20	2.20	2.41	1.55	1.65
462-5-1	1.64	• 50	.70	1.00	1.00	4.84	1,55	• 80
462-5-2	1.64	.50	.70	3.00	3.00	.79	1,55	2.18
462-5-3	1,25	• 56	.80	3.00	3.00	.79	1,55	2.18
462-5-4	1.25	• 56	.80	1.00	1.00	4.84	1.55	• 80
75907-11-1	1.64	.50	.70	2.20	2.20	2.41	1.55	1,65
75907-11-3	1.64	.50	.70	2.20	2.20	2.41	1,55	1.65
75907-11-5	1.64	• 50	.70	2.20	2.20	2.41	1.55	1.65
75907-11-2*	2.75	• 50	.70	• 86	• 86	3,22	4.06	1.03
75907-11-4	2.75	• 50	.70	•86	• 86	3.22	4.06	1.03
75907-11-6	2,75	• 50	.70	• 86	•86	3.22	4.06	1.03

^{*11-2, 11-4, 11-6 -} Phase II Formulation

^{**} Figure II, March 1987 monthly report, Phase III

FIGURE V* - FREEZE/THAW STABILITY - % SEPARATION

Formulation	1 Cycle	2 Cycle	3 Cycle	4 Cycle	5 Cycle	6 Cycle	7 Cycle	8 Cycle
462-4-1	11	<b>}</b>	<b>}</b>	<b> </b>	⊢	۰	<b>-</b> -	
462-4-2	0	0	0	<b>-</b>	0	<b>-</b>	0	
462-4-3	0	0	0	<b>-</b>	<b>-</b> -	-		
462-4-4	0	0	0	0	0	<b>}</b>	0	
462-5-1	က	15	14	21	ı	1		
462-5-2	<b>-</b>	<b>-</b>	0	<b>-</b>	0			
462-5-3	0	<b>-</b>	0	<b> </b>	0			
462-5-4	27	1	ı	1	ı	t	ı	
75907-11-1	<b>—</b>	<b>-</b>	1-	<b> </b>	F	H	0	
75907-11-3	0	<b> </b>	0	0	H	<b>-</b>	<b>}</b>	<b>-</b>
75907-11-5	0	<b>—</b>	<b> </b>	<b>-</b>	<b>-</b>	<b>-</b>	0	
75907-11-2	4	8	19	19	19	21	ı	
75907-11-4	4	9	15	15	17	19	ŧ	,
75907-11-6	4	9	15	16	24	ŧ	1	

^l - Trace - Not Measurable

* Figure III, March 1987 monthly report, Phase IIÎ

## FIGURE TV 72

#### Restricted

C.A. Sterling - Personal Care Products - 230-25-06

N. A. Randen - Person Care Products - 230-25-06

From: F. D. Griffith - Medical, Toxicology Services - 230-25-06

Subject: Potential Toxicity of Arthropod Repellent Formulations

Date. April 2, 1987 (Formulation Corrected June 9, 1987)



It is my opinion that the differences between Formula 1 and Formula 2, below, are toxicologically insignificant.

The changes are small ratio differences of minor constituents which are common cosmetic ingredients.

Ingredient	Formula 1	Formula 2
Cabosil M-5	2.75	1.64
Varonic L1420	0.65	0.65
Varonic L148	1.03	1.65
Lexemul	4.06	1.55
Carbowax 400	0.98	0.98
Liponic EG-7	2.26	2.26
Veegum	0.70	0.70
Natrasol 250H	0.50	0.50
Lexol PG 865	3.22	2.41
Waxenol 816	0.65	0.65
Arlamol E	0.86	2.20
Adol 63	0.86	2.20
Polymer *	5.83	5.83
DEET *	35.00	35.00
Germaben II	0.22	0.22
Water	To 100%	To 100%

FDG/MAC FDG:bh (TS84 2.6)

#### Restricted

XX Figure II, march 1987 monthly Report, Phan III

^{*} Corrections

Personal Care Products
Consumer Specialties Division'3M

3M Center St. Paul, Minnesota 55144-1000 612/733 1110



July 20, 1987

Dr. Arturo Castillo
Environmental Protection Agency
Registration Division (TS-767C)
Insecticide-Rodenticide Branch/PM Team 17
401 M Street S.W.
Washington, DC 20460

Dear Dr. Castillo,

Consumer Specialties Division/3M has recently filed an application with EPA for the registration of our Insect/Arthropod Repellent Lotion. In response we have received an EPA File Symbol/Registration No-58007-R. In addition we have received a "Report of Analysis for Compliance with PR Notice 86-5" dated June 3, 1987. While the noted corrections stated in the letter were suppose to accompany "future data submissions", we thought it prudent to submit them now.

Firstly, new compliance with Good Laboratory Practices Standards statements have been obtained. These were signed after the completion of the studies as opposed to those in our original application which stated that the GLP standards would be followed. Three copies of the following statements are enclosed:

MRID 40214902 Volume 3 3M's Insect/Arthropod Repellent Lotion (35% DEET) Acute Oral Toxicity - Rats 3M 60405110-00003.

MRID 40214903 Volume 4 3M's Insect/Arthropod Repellent Lotion (35% DEET) Acute Dermal Toxicity 3M 60405111-00003.

MRID 40214904 Volume 5 3M's Insect/Arthropod Repellent Lotion (35% DEET) Primary Eye Irritation - Rabbit 3M 60405113-00003 and 3M 60405113-00064.

MRID 40214905 Volume 6 3M's Insect/Arthropod Repellent Lotion (35% DEET) Primary Dermal Irritation 3M 60405112-0003

Dr. Arturo Castillo July 20, 1987 Page 2

Secondly, the Data Requirement on the title page of MRID 40214901 Volume 2 - Product Chemistry Data Requirements for 3M's Insect /Arthropod Repellant Lotion has been changed from 40 CFR Part 158.120 to Guideline Numbers 61, 62, 63 as requested.

Thirdly, the legibility of some of the material in MRID's 40214902, 40214903, 40214904, 40214905, and 40214906 was reported as marginal. The only page I could see which was hard to read was the assay of the active for the formulation, attached as page 3 of 3 in the confidential section of these studies. This assay has been re-run using black ink instead of blue. Three copies for each study are attached.

Fourthly, the only comment not addressed herein is for MRID 40214904 wherein a repeated washed primary eye irritation study was attached to the original study. It was stated that these should have been bound as separate studies. If this is a major problem I can go ahead and do this and resubmit these as separate studies. Please let me know.

And finally, the letter attachment to the letter to Mr. Tauano comparing 3M's Phase III formulation with the Phase II formulation has been corrected to show that there is only 5.83% acylate polymer in the formulations and not 23.33% as shown. Three copies of the correct form are attached.

If I can be of further assistance please let me know.

Sincerely,

Neil A. Randen Research Specialist

NAR/cab

**Enclosures** 

Figure VIII

### PERCENT SEPARATION VIA FREEZE/THAW CYCLING

<u>Formulation</u>	Fre	eze/Th	aw Cy	cle Nu	mber		
•					•		
	1	<u>2</u>	. <u>3</u>	4	<u>5</u>	<u>6</u>	<u>7</u>
CHP7 (Pilot Batch)	o .	٥.	0	0	0	0	0
CPP7 (Production  Trial Batch)	2.9	2.2	3.4	4.0	4.1	4.0	3 <b>.</b> 7
Phase II Formulations*	4.0	7.0	16.3	16.7	20.0	22.0	
Laboratory Phase III**	0	.1	0	0	.1	.1	0

^{*} For comparison

^{**} For comparison, Laboratory batch with same composition as production batches. March Monthly Report.

# FIGURE IX*

## DISPENSIBILITY

<u>Formulation</u>	Container	% Dispensed
Phase I	LDPE	89.2%
Phase I	"Cutter's" Bottle	88.7%
Phase I	"Off" Bottle	88.6%
Phase II	HDPE	.88.9 <u>+</u> 2.5% n=5
Phase II	LDPE	92.1%

^{*} Figure V, March 1987 monthly report, Phase III

FIGURE X

# STABILITY CHART

Aesthetic Value		III Contract	
DEET Analysis		nsion to Phase	
% Weight Loss	Contract	Testing included in Proposed Six Month Extension to Phase	evised Proposal
Viscosity	Testing part of Phase III Contract	cluded in Propos	Testing included in this Revised Proposal
됩	Testing pa	Testing in	Testing inc
% Separation			
Time Interval	Zero time 1 month 1 month	3 months 3 months 6 months 6 months	<ol> <li>year</li> <li>years</li> <li>years</li> <li>years</li> <li>years</li> </ol>
Test Condition	Initial 113°F Room Temp	20°F 113°F Room Temp 113°F Room Temp	Room Temp Room Temp Room Temp Room Temp

5.2 Appendix B

THE 38 INSERT/AINTEROND SEPTILEUT LOTION  WIN 2016. (394 L.)  FORM A MASSURE Cross  CHB /  FORM A MASSURE Cross  CHB /  MARTE OVER  MARTE	FORM 12	24 iRev 11/8	6)	PILOT BATCH	GE 1 of 6	WALGREEN L			. JJ 5. INC	,
CHB 7  DAY, JOHN 6-30-87  SUPERSSOL SY, CHARGEL LIM DAY, DAY, TOPEN CHICKEN BY, CHARGEL LIM SUPERSSOL SY, CHARGEL LIM DAY, DAY, TOPEN CHICKED BY, CHARGEL LIM DAY, TOPEN CHICKED BY, CHARGEL LIM TOPEN CHICK LIM TOPEN CHICKED BY, CHARGEL LIM TOPEN CHICKED BY, CHARGE LIM TOPEN CHICKED BY,	TITLE	3M INSEC	T/ARTIRO	POD REPELLENT LOTION WP	N 20186					
DAT - POPID 5-30-67						_				
DATE TO STATE AND DATE TO STAT			•		,		CHE	17		
APPLOVED NY.  ALTHOUGH NY.  AND WARD INGSEDENTS  ORDER NO.  AND WARD INGSEDENTS  OUANIITY  DISTORMULA NO.  SERIALA NO.  CONTROL  AND WARD  DISTORMULA NO.  SERIALA NO.  CONTROL  OUANIITY  NORTH.  CONTROL  CONTROL  CONTROL  CONTROL  NORTH.  NORTH.  CONTROL  NORTH.  NORTH.  NORTH.  NORTH.  NORTH.  CONTROL  NORTH.  NORTH.  CONTROL  NORTH.  N	ś			/ · V f B / I i i s	, 7					
REPRODUCTION CHICGED BY.  LOTTE, MANUAL OATE, 7-1-87 STEAL NO.  COOK NO.  AMOUNT FORMULA NO.  FORMULA NO.  SOUND NO.  SOUND MASS CCD ADD NO.  BETT 8 TIDE BOTT STEPTET TO THE ACC.  PART 1: Place in a 60 gallon foren till kettin county of the propellar type size:  Rectle Bacd: It 1 - (1) Careta.  Ricer Board Tibe a propellar type size:  Rectle Bacd: It 1 - (1) Careta.  Ricer Board Tibe Address Stepted Tibe Acc.  Ricer Board Tibe Acc.  Ricer			$c \rightarrow 1$		.87	MAT'L O.K.				
S VIV WPN BASEDENIS OUANIHIT COMPAND ASSO CCO ADD UNDER SY BY	•	- •		Parth allarge Dure 7-	4-87	ALC. %				
S V/N   WPN						CODE NO.				
S. V. WIN   MARKET TIME SELECTION   MARKET   M		·		AMOUNT - PORMOIA NO.		<del></del>	T	1 - 2 <i>1</i>	7156	
PRET I: Place in a 60 gallon Groen tilt kettle  PART I: Place in a 60 gallon Groen tilt kettle  equipped with a propeller type sizer:  Kettle Badd:		≴ ¥/¥	WPN	INGREDIENTS	QUANTITY	CONTROL	,	1 .	J	1
equipped with a propeller type miner:  Kettle Used: #1   PUR Annea    Rizor Used:   Checked for Cleanianes By Manna    Bate & Time Added:   WOVE TIME    Bate & Time Added:   WOVE TIME    Bate & Time Added:   WOVE TIME    Joseph   Date & Time Added:   WOVE TIME    Bate & Time Added:   WOVE TIME    Joseph   Date & Time Added:   WOVE TIME    Joseph   Date & Time Added:   WOVE TIME    Joseph   Date & Time Added   WOVE TIME    Joseph   Date & Time Added   WOVE TIME    Joseph   Date & Time Added   Date    Joseph   Date   Date    Joseph   Date & Time Addition Storted:   J. S. P. P. P.    Joseph   Date & Time Addition Storted:   J. S. P. P. P.    Joseph   Date & Time Addition Storted:   J. S. P. P. P.    Joseph   Date & Time Addition Storted:   J. S. P. P. P.    Joseph   Date & Time Addition Storted:   J. J. P. P.    Joseph   Date & Time Addition Storted:   J. J. P. P.    Joseph   Date & Time Addition Completed:   J. J. P. P.    Joseph   Date & Time Addition Completed:   J. J. P. P.    Joseph   Date & Time Addition Completed:   J. J. P. P.    Joseph   Date & Time Institute Completed:   J. J. P. P.    Joseph   Date & Time Institute Completed:   J. J. P. P.    Joseph   Date & Time Institute Completed:   J. J. P. P.    Joseph   Date & Time Institute Completed:   J. J. P. P.    Joseph   Date & Time Institute Completed:   J. J. P. P.    Joseph   Date & Time Institute Completed:   J. J. P. P.    Joseph   Date & Time Institute Completed:   J. J. P. P.    Joseph   Date & Time Institute Completed:   J. J. P. P.    Joseph   Date & Time Institute Completed:   J. J. P. P.    Joseph   Date & Time Institute Completed:   J. J. P. P.    Joseph   Date & Time Institute Completed:   J. J. P. P.    Joseph   Date & Time Institute Completed:   J. J. P. P.    Joseph   Date & Time Institute Completed:   J. J. P. P.    Joseph   Date & Time Institute Completed:				Date & Timo Batch Started: 1-7-87 104	Am	NUMBER	BY	BY	L BY	土
Kettle Bacd: # Till Anger  Mixer Bacd: # Till Anger  Oncolog for Cleaning By: Manager  Date & Fisc. 7-R 7 10:50   Anger  Manager Manager  And Manager Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager  Manager							上		上	上
								<del> </del>	<del> </del>	$\pm$
Date 8 Time: 1.7-8.7   10:50   Ann				Mixer Used: Cal			<del> </del>	<del> </del>		F
Pato & Time Added:							$\vdash$		_	F
25.95   105310   Purified Water, U.S.P./H.F.   143.8 Kg.   MAI   SULFH   SUL							<del> </del>	<del>                                     </del>	<u> </u>	F
Add without sixing, to the water: (36 cal.)   Add without sixing, to the water: (36 cal.)   Add without sixing, to the water: (36 cal.)   Add without sixing, to the water   (36 cal.)   Add without sixing without sixing without   (36 cal.)   Add without sixing without   (37 cal.)		35 05	105310		143 8 Kg		1111	60	111	Ļ
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Solve tort to mix.   Simple	7/7/27	37	Toini	Dale & Time, Addition Stantid! 7-7 87 11-05 A PA			10,1		111	Ļ
Mash the walls of the kettle with a small support of Purified Vator, U.S.F./R.F. (VFR 10531c).	, <del>-448/</del>	1000		1 Daletine Adahan lendelili 7-7 17 1120A	M	- C.00161	W.	<u>&gt;w</u>	FIR	2
manual of Purified Vator, U.S.F./R.F. (VFR   105310).   105310).   105310).	7(	297/7/87	<b>h</b>							L
Increase the speed of the propeller mixer so that the batch is mixing thoroughly.  Add while mixing:  Date & Time Addition Started: 7 7-87 H:35AAA  2.26 11318 Polyethylene Glycol (7) Glyceryl Ether 9.040 Kg. CEGIGT MASW.HAS (Liponic EG-7)  0.98 10384 Polyethylene Glycol (8) (Garbowns 400) 7.920 Kg. CEGIGT HASW.HAS 0.7 12929 Magnesium Aluminum Silicate (Yeerum) 2.800 Kg. CEGITAL HASW.HAS 1.00 Mg. CEGITAL HASW.HAS 1.00 Mg. CEGITAL HASW.HAS 1.00 Mg. CEGITAL HASW.HASW.HASW.HASW.HASW.HASW.HASW.HASW.		Murder	·	amount of Purified Water, U.S.P./N.F. (WPR		_				
that the batch is mixing thoroughly.  Add while mixing:  Date & Time Addition Started: 7 7-87   11:35 And    2.26   11318   Polyethylene Glycol (7) Glyceryl Ether   9.040 Kg.   880767   MA SW. MA    (Liponic EC-7)  0.98   10384   Polyethylene Glycol (8) (Carboway 400)   3.920 Kg.   C785767   MH SW. MA    0.7   12929   Magnesium Aluminum Silicate (Yoccum)   2.800 Kg.   C785767   MH SW. MH      Date & Time Addition Completed: 7.7-87   11:45 And     Mow heat Part I to 480 - 500 C.     maintaining the temperature no higher than   Candidate     500 C.   while mixing.     Date & Time Heating Started: 7-7-87 - 2007   Candidate     Date & Time Heating Completed: 7.7-87 - 2007   Candidate     Date & Time Heating Completed: 7.7-87 - 2007   Candidate     Date & Time Heating Completed: 7.7-87 - 2007   Candidate     Date & Time Heating Completed: 7.7-87 - 2007   Candidate     Date & Time Heating Completed: 7.7-87 - 2007   Candidate     Date & Time Heating Completed: 7.7-87 - 2007   Candidate     Date & Time Heating Completed: 7.7-87 - 2007   Candidate     Date & Time Heating Completed: 7.7-87 - 2007   Candidate     Date & Time Heating Completed: 7.7-87 - 2007   Candidate     Date & Time Heating Completed: 7.7-87 - 2007   Candidate     Date & Time Heating Completed: 7.7-87 - 2007   Candidate     Date & Time Heating Completed: 7.7-87 - 2007   Candidate     Date & Time Heating Completed: 7.7-87 - 2007   Candidate     Date & Time Heating Completed: 7.7-87 - 2007   Candidate     Date & Time Heating Completed: 7.7-87 - 2007   Candidate     Date & Time Heating Completed: 7.7-87 - 2007   Candidate     Date & Time Heating Completed: 7.7-87 - 2007   Candidate     Date & Time Heating Completed: 7.7-87 - 2007   Candidate     Date & Time Heating Completed: 7.7-87 - 2007   Candidate     Date & Time Heating Completed: 7.7-87 - 2007   Candidate     Date & Time Heating Completed: 7.7-87 - 2007   Candidate     Date & Time Heating Completed: 7.7-87 - 2007   Candidate     Date & Time Heating Completed: 7.7-87 - 2007   Candidate     Date & Time										上
Date & Time Addition Started: 7 7-87 II.35.401  2.26 11318 Polyethylene Civcol (7) Civcervi Ether 9.040 Kg. CECOLGT HIM SW. HIM S. (Liponic EG-7)  Q.98 10384 Polyethylene Civcol (8) (Carbonny 400) 3.920 Kg. C785767 HIM SW. HIM S. O.7 12929 Hagnesium Aluminum Silicate (Veccum) 2.800 Kg. C785767 HIM SW. HIM S. Date & Time Addition Completed: 7.7-87 III.45 Aca Hov heat Port I to 480 - 500 C. III.45 Aca Hov heat Port I to 480 - 500 C. maintaining the temperature mo higher than 5.00 C. while mixing.  Date & Time Heating Started: 7.7-87 - 2000 C. Mills mixing. The Heating Completed: 7.7-87 - 2000 C. Mills mixing. The Pinal Temperature Attained: 100 C. Mills Mixing Maintain Port I at 480 - 500 C. with constant mixing until ready to be added to Part II.  PART II: Place in a 120 gallon Green keitle gauf prod With Newson action mixing and adde nersponen.  Kettle Uncd. Track-rate Chacked for Clean incasing: Army Mixing Mixi										F
2.26 11318 Polyethylene Clycol (7) Clyceryl Ether 9.040 Kg. CSC0767 NN SW. NN S (Lipenic EG-7)  0.98 10384 Polyethylene Clycol (8) (Carbowns 400) 3.920 Kg. C785767 NN SW. NN S 0.7 12929 Magnesium Aluminum Silicate (Yaccum) 2.800 Kg. (781767 NN SW. NN S 0.7 12929 Magnesium Aluminum Silicate (Yaccum) 2.800 Kg. (781767 NN SW.				Add while mixing:						F
O.98 10384 Polyethylone Glycol (8) (Carbowax 400)  O.7 12929 Magnesium Aluminum Silicate (Vaccum)  Date & Time Addition Completed: 7.7-87 U.45 Am  Now heat Part I to 40° - 50° C.  maintaining the temperature no higher than  50° C. while mixing.  Date & Time Heating Started: 7-7-87 - 2007 Samula by the Carbowax  Date & Time Heating Completed: 7.7-87 - 2007 Samula by the Carbowax  Date & Time Heating Completed: 7.7-87 - 2007 Samula by the Manual Tolor Panal Temperature Attained:  Maintain Part I at 40° - 50° C. with  constant mixing until ready to be added to.  Part II.  PANT II: Place in a 120 gallon Green kettle  gaulped with swarp action mixing and pide  Octooked for Clangliness by Army Army  Chacked for Clangliness by Army  Date & Thur: 3 - 3 - 3 - 3 - 3 - 3 - 3 - 3 - 3 - 3				Date & Time Addition Started: 7 7-87	11:35AM	<b></b>				_
Date & Time Heating Started: 7-7-87 2000 Manual April 11: Place in a 120 gallon Groen kettle gaugepad with never action mixing and oide.  Pattle Uned: 7-7-87 11: 45 AM  Now heat Port I to 480 - 500 C.  **Simulation as W. In Careta Completed: 7-7-87 - 2000 Manual April 11: Place in a 120 gallon Groen kettle gaugepad with never action mixing and oide.  **Stile Uned: 7-7-87-1-1-1000 Manual April 11: Place in a 120 gallon Groen kettle gaugepad with never action mixing and oide.  **Chacked for Clannings in the constant of the		2.26	11318		9.040 Kg.	C866T67	妣	SW.	此	S
Date & Time Addition Completed: 7.7-87 11:45 AM  Now heat Part I to 480 - 500 C.  maintaining the temperature no higher than  500 C. while mixing.  Date & Time Heating Started: 7-7-87 - 2007 Amelia of the Lucylest Date & Time Heating Completed: 7.7-87 - 2007 Amelia of the Lucylest Date & Time Heating Completed: 7.7-87 - 2007 Amelia of the Lucylest Date & Time Heating Completed: 7.7-87 - 2007 Amelia of the Lucylest Date & Time Heating Completed: 7.7-87 - 2007 Amelia of the Lucylest Date & Time I at 400 - 500 C. with constant mixing until ready to be added to Part II.  PART II: Place in a 120 gallon Green kettle square and part of the Startest Date of Time Kernic Amelia of Time Amelia of Time Kernic Amelia of Time Kernic Amelia of Time Ameli		0.98	10384	Polyethylone Glycol (8) (Carbownx 400)	3.920 Kg.	C785767	妣	3W	出出	3
Now heat Part I to 480 - 500 C.  Now heat Part I to 480 - 500 C.  Maintaining the temperature no higher than  500 C. while mixing.  Date & Time Heating Started: 7-7-87 - 2 2011  Date & Time Heating Completed: 7-7-87 - 2 2011  Pant I complete Time Heating Completed: 7-7-87 - 2 2011  Maintain Part I at 400 - 500 C. with  Constant mixing until ready to be added to  Part II.  PANT II: Place in a 120 gallon Groon kettle  January There  Sanding all Maintain for the first than the first t		0.7	12929	Magnesium Aluminum Silicate (Veccum)	2.800 Kg.	1281767	111	5.W	地	3
maintaining the temperature no higher than  50° C, while mixing.  Date & Time Heating Started: 7-7-87-2007  Date & Time Heating Completed: 7-7-87-2007  Date & Time Heating Completed: 7-7-87-2007  Pinal Temperature Attained:  Maintain Part I at 40°-50° C, with  constant mixing until ready to be added to  Part II.  PART II: Place in a 120 gallon Groen kettle  ugui pped with sweep action mixing and oide  acropora:  Kettle Uncd: 1/ Terkenic				, Date & Time Addition Completed: 7-7-87	11:45 AM					_
Date & Time Heating Started: 7-7-87-2007  Date & Time Heating Completed: 7-7-87-2007  Date & Time Heating Completed: 7-7-87-2007  Pinal Temperature Attained:  Maintain Part I at 400-500 C. with  constant mixing until ready to be added to  Part II.  PART II: Place in a 120 gallon Groen kettle  causing part it as a second and added to  causing part it as a second and added to  causing part it as a second and added to  causing part it as a second and a second					¥ Sim		Cur	Eul V	rint	- ~
Date & Time Meating Completed: 7.7-57-2.08-4  Pinal Temperature Attained: 400 - 500 C. with  constant mixing until ready to be added to  Part II.  PART II: Place in a 120 gallon Groen kettle  uguipped with sweep action mixing and oide  acrapara:  Kettle Uned A. 7 terkerate  Chacked for Clandiness By: Always Always  Date & Time: 17-68-762  Essent					11 Saisk	Shart Port	edu	e		-
Date & Time Meating Completed: 7.7-57-2.08-4  Pinal Temperature Attained: 400 - 500 C. with  Constant mixing until ready to be added to  Part II.  PART II: Place in a 120 gallon Groen kettle  Under the second of				Date & Time Heating Started: 7-7-87-2-	Sulti	41		THE	33	ı
Maintain Part I at 40° - 50° C. with  constant mixing until ready to be added to.  Part II.  PART II: Place in a 120 gallon Groon kettla  gauipped with aware action mixing and side  agrapara:  Kettle Uned A 7 7 or Kernel  Chacked for Claimliness By: Arrogarine				Date & Time Heating Completed: 7-7-87 -24	0 200	We hold 27		7/7/87		
PART II: Place in a 120 gullon Groon kettle  gauipped with aware action mixing and side  neropora:  Kettle Uned A 7/cr Kerice  Chacked for Claunlineau By: Amore Amore  Pate A Time: 1276 8:3976						1 22	911	GA	W	:
PART II: Place in a 120 gullon Groon kettle  gauipped with aware action mixing and side  neropora:  Kettle Uned A 7/cr Kerice  Chacked for Claunlineau By: Amore Amore  Pate A Time: 1276 8:3976				constant mixing until ready to be added to		1000	糊	7	7=3	
Kettle Uned Al Tier Kernic Chacked for Claim incoming and oide  Chacked for Claim incom by: Almortanic Chacked for Claim income by: Almortanic Chacked for Chacked					11-11-11-11-11-11-11-11-11-11-11-11-11-	h 1	<u>//</u> /	二	二	
Kettle Uned A / Tier Kernic Chacked for Claunlineau By: Almortamed	<u>'</u>			agui prod with aveen action mixing and nide	H= Horama Longin		#	二	二	
Pate A Time: 1776 8:39m				,	<del>(-)</del>		士	士		
NOTICE: ANY PERSON INITIALING WORK DOWN MILL ALLY THEIR SIGNATURE TO FITHER THIS PAGE OF THE LAST PAGE				Chacked for Claunliness by: A trop from	<u></u>		士	士	$\exists$	
		NOTICE	. ¥WA ₩€t	SON INITIALING WORK DOWN MICH ACTO THE CICHATHER TO	FITHER THIS PAGE OF TH	IF IAST PAGE				

= JORH_122	4 [Rev. 11/6	6)	PILUI BAIGH ,	AGE 2 of 6	WALGREEN L		TORIE	S, INC	<u>.</u>
1	***	-		20186 N	CONTROL NO.				
C MUITY	400 Xg.	(394 L.)	FORM & MEASURE Cream		(	CHB	7		
10C Ad			SUPERSEDES	<u>'</u> , L					
IS ISS EY.			tridil Jijj DATE: 7/6/8	<u>/</u>					
ALAFPROYED	8Y:	let.	Try DATE: 7-6	-87	MAT'L O.K. ALC. REQ.				_
REPRODUCT			da DATE: 7	-6-87	ALC. % SERIAL NO.				_
0			AMOUNTFORMULA NO		CODE NO.				_
	\$ ¥/¥	WPN	INGREDIENTS	QUANTITY	INGRED. CONTROL NUMBER	MSRD	CKD	ADD 8Y	
		<u> </u>	usie a lieu kacition Stafted: 9:514 111	.e <del>7</del>	NOA.PEK	<u>"</u>			t
_				į į		1,11		1	ł
	26.92	12095	65:1.5:1.5 Edle fatto iso-Octyl Acrylate:Steeryl Kethecrylate:Acrylic Acid	107.7 κε.	C559167	TAN	Sw	144	T
_ *	<b>5</b>		Terpolycer-21.05% in Deet				<u> </u>	二	İ
- See DOCKE	13.91	10554	N,N-Diethyl-F-Toluctice (MCK Diethyltoluccide	55.6 kg.	C74167	烘坩	521	1111	ł
- part	616	<del> </del>	95% Mota Isozer Hinimus)			-	<del>  `</del>	<del> </del>	1
- Julium	(ZD ISM		Date & Time Addition Completed: ][] [3] 10:5	ИŁ					Į
- "//	44.700.44	4	Begin to mix Part II with sweep action mixing		1		二		ţ
	7/11/57	<u> </u>	and side scrapers at a slow speed.			-	<del> </del>	}	ŀ
-	1	<b> </b>	Sweep Action Mixer Speed Setting: 6.5		]				ŗ
			Heat Part II to 59° - 61° C. maintaining the temperature no higher than 61° C. while						Ľ
11 - i		1	mixing.		<u> </u>				_
<u>"</u>	<del></del>	<del> </del>	Dute & Time Heating Started: [:05PM 7/16]		<del> </del>	-	<del>  </del>		_
			Date & Time Meating Completed: 77/67 J. 20Ph Final Temporature Attained: 68°C						F
			Add carefully while mixing:		<b></b>				<u> </u>
			Date & Time Addition Started: 7 1127 1:2514			-			F
	1.65	12931	Polyethylene Glycol (82) Glyceryl Monotallowate (Varonic L148)	6.600 Fg	C775161	批	Skr	HH	Ē
	0.65	12930	Polyethylene Glycol (200) Glyceryl	0.500.11	C796767	UU	5/15	1111	-
			Honotallowate (Varonic L1420)	2.600 Kg	1.7.80.07	/111	200	ACI	_
	1.55	11317	Clyceryl Stearate/Sodium Lauryl Sulfate (Lexemul AS)	6.200 Kg	C784T67	地	SW	UR	- -
	2.41	11315	Propylene Clycol Dicaprylate/Dicaprate (Lexol PG865)	9.640 Kg.	C183767	加	3W	HZ	- -
	0.65	13000	Catyl Palmitate (Waxenol 816)	2.600 Kg.	C793167	SP .	ساک	HC	-
,	2.2	10026	Polypropylene Glycol (15) Stearyl Ether	8.800 Kg.	C811767	18	5W	111-	-
		3	(Arlamol E)		ļ				-
	2.2	10046	Cetyl Stearyl Alcohol (Adol 63)	8.800 Kg	CHIGI,	MA.	SW.		3
			Date & Time Addition Completed: 7/7/87 -2	) d A41	60				-
			Maintaining the temperature of Part What	~ T	191115	145/	767	$\rightrightarrows$	
	115/11	1	590 - 610 C., mix until all the added ingredients are completely dissolved and mixed		of Sour	//+// //	27		
	IJAN (JA)	J	unit'ormly.	ý.	The state of the s		4	$\dashv$	
		T	Date & Time Part II Completely Dissolved						
(3/10)	3-3411	<del>/</del>	and Uniform;	n A	11/10				
Y(1)-1-1	7	1 4 11 2 2 2	DOOR HISTALING WOOV COLE HAVE	Nex-North	ymell!				:
	NUTICE	C ANT PE	RSON INITIALING WORK DONE, MUST AFFIX THEIR SIGNATURE TO	D EITHER THIS PAGE OR T	HE LAST PAGE.				

Add WPN 12098 directly into 120 Gallon Groen Kettle. Now using same container which was used to weigh 12098, weigh out WPN 10534, making swe to mix & rinse container well.

Polist O 2 Juillab (34.06)

> Stare Wonderdo 7/7/87

OIH 122	4 (Rev. 11/86	51	PILOT BATCH ,		3 of 6	٠.				- 36	<u> </u>
	<u></u>	<u></u>					ALGREEN L		CIOXIE	S, INC	
			OD REPELLENT LOTION W	PN	20186		•				
31	400 K _F . (	394 L.)	FORM & MEASURE Cross				U	HB.	7		
	IED <u>6-30</u>		5 197 K 11/18	,	Ĺ						
			MIZUII . DATE: 7/6/8		<del></del>						
PROYED	BY:	lety	Try DATE: 7-6	-87	7 	M.	AT'L O.K. LC. REO.				
	IIOH CHECKE	- 11	de DATE: 7		<b>~</b> -	A					
					•	CC	DDE NO				
ADER NO.	·		AMOUNT FORMULA NO.		<del></del>	₩, ——	.O. NO		<del></del>		
	≴ ¥/¥	WPN	INGREDIENTS	.	QUANTITY	-	INGRED.				1 .
		l		+	K c c	$\dashv$	NUMBER	BY		ay .	<del>1-</del>
	V-71		Heat Part II to 810 - 850 C. along with coderate agitation maintaining the temperature	Ι,	Sample as b	ZV	be septe	12	Trem	in	二
	1. V. h. h.	1.1	'no higher than 63' C. for not acro than 5	1/	Zalusting	13	D. Bar	<del></del>	+ 211	less	Im
L'st		7/1/27	-ainutes: ¥	<del></del>		DI	7	7	7-5	1	
	my 7-6-		Sweep Action Mixer Speed Setting: /			7 1	Steel	7	With	1	حهل
	0		Date & Time Heating Started: 7/7/87- 2/5	to t	Saute -	afr.	7/4.0	1-/	10 10	1-4-5	12
	Shall Sh	7-27	Date & Time Heating Completed 7/7/87 - 230	1-1	- 110	X - L		$\Box$	yw		Ī
- 4/3	Shall F	[7] E/T-	Final Temperature Attained: 8/0 C. Part I Compiley district of limiting. 71787 2:4	J		, d	學言	V	7	Y	厂
M.	the Gath	7-11-6	Add slowly to Part II while caxing:	Thu-			4117	┼	<del> </del>	├	╁╌
10/01				<del>                                     </del>		寸	<del>-44°/-</del>	<del>                                     </del>	1	<del>                                     </del>	十
<i>!!!</i>			Date & Time Addition Started: 2/7/87 2"	15 PM		耳		$\sqsubseteq$	$\Box$		匚
	0.5	11616	Collulose, 2-Hydroxy Ethyl Ether (Hetrasol 250	┼	2,000 Kg.	-1;	C782167	766	1200	20	1
		-	Hill)			<u>J`</u>	-106101	V Tyre		700	17
	<del> </del>	}	Weta A Pica Addition Company - Information	120		7		$\sqsubseteq$	lacksquare		$\sqsubset$
,. <del>.</del>	<del> </del>	<del> </del>	Date & Tizo Addition Completed: 7/7/37 2.5	h132							<del> </del>
1	.5/10		Hix Part II until a good dispersion is	<del>                                     </del>		十			<b> </b>	<del>                                     </del>	-
1 W		167	obtained, about 30 zzaules. 15 minutes			二					
7-7-1	7 10/	//8/	Mixing fice: 250 pm to 305 pm	<del> </del>				<del> </del>	<del> </del>		├-
		<del>'</del> -		<del> </del>		- -			<del> </del> -		<del> </del>
			Date & Time Part II in Uniformly and Well Dispersed: 7/7/87 3:10 PM			王					E
			Increase Part II mixing to give rapid	<del> </del>	<del></del>	4		<u> </u>			
			agitation.	<del> </del>		+			<del> </del>		<del> </del> -
						土					
			Sweep Action Mixor Speed Setting: /#	<del> </del>							
			With Part II at 810 - 850 C. slowly add	<del> </del>		+		<del> </del>			<del> </del>
			Part I.			ユ					
			Rahout Part II if necessary to 810 - 830 C.	<del> </del>		+			<b> </b>		-
			included the second sec	<del> </del>		+		<b> </b>			<del> </del>
			Initial Temperature: 8/°C			工					
			Dulo & Time Reheating if Necessary Started: Not Necessary	<del> </del>		- -			<b>  </b>		_
			Dute & Time Relieuting if Mecessary	<del> </del>		-{-					<b>-</b>
			Completed: Not Necessary	1.0						=	
			Final Temperature Attained: (Iftallel of	yly		- -			<b></b>		<b></b> ,
			Date & Time Part I Addition Started: 7/7/87, 6	1029		╬	<del>/</del> -				<b>-</b>
			Date & Time Part I Addition Completed: 7/1/23	22/4	7 .	土	10				
			Para Para Para Para Para Para Para Para	<b>/</b> *	Ambal Kunter	梁	; OVL				
			Ringe Part I kettle with a small amount (less than I gallon) of Purified Water, U.S.P./N.F.	(464	eat Contined	. F	U 747	6-31 6-31	1-x	20-	_
			(WPN 10531C) and add the rinsings to the batch	35	Tested! 7-7-	37	7 3/ AK	7			,
			(combined Parts I & II).		19The Robe		s 20 Mes	Bes	2	$\Box$	<del>_</del> ,
			Mix the batch for 20 minutes at 810 - 830		white T		17/2 343		200		
<b>D</b>	<del></del>		c.	120	BAL HEWALL	-14	amed!			<del></del>	<del>.</del> .
			.,			17	GIL L	2///		KILL	上
			Mixing Time: 345 PM to 405 PM			K	FIGURA			华丁	<del>-</del> -
	1		• • • • • • • • • • • • • • • • • • • •	i		ſ	<u> </u>	5/70	n 1	- 101	₹ ,
			<del></del>	ļ		-	<del>//</del>	<del>}/\\/</del> 1	<del>/</del> +		

ORM-	1224 (Rov. 11/8	6]	PILOT BATCH PA	GE 1 of 6	WALGREEN L	ABORA	TORIE	~~;, 5. INC	, :
TITLE	3M INSEC	r/ala diore		พ20186	CONTROL NO.				
			FORM & MEASURE Cream	<del></del>					
•	OPTED 6-30	1	- <del></del>				•		
į	-28160 <u>- 0-70</u>		・ <i>リルト はは</i> カノルノカコ	•	<u> </u>				
ISSUE	·	100	DATE: //6/8/	0-7	MAT'L O.K.				
APPROY	ED BY	XILOI	f Try DATE: 1-6	<del>-8</del> /	ALC. REQ. ALC. %				
REPROD	NCIION CHECKE	D BY.	DATE:	7-6-81	SERIAL NO.				
ORDER	но		AMOUNT FORMULA NO		W.O. NO.				
	X ¥/¥	WPN	INGREDIENTS	YTITHAUO	INGRED. CONTRÔL NUMBER	MSRD BY	1	ADD BY	SKI CKI
	<del></del>		Force cool the batch with mixing to 370 -	<del> </del>		<del> </del>	<del>  </del>	<u> </u>	
			39° C.			<del></del>	$\sqsubseteq$		尸
			Adjust the mixing speed while force cooling to						二
			entrapment of air.	·		<del> </del>	-		
			10			<del> </del>			$\vdash$
			Sweep Action Mixer Speed Setting: / 4			#=			二
			Initial Temperature Before Force Cooling: 8/°C			<del> </del>	<del> </del>	-	-
			Date & Time Force Cooling Started: 7-7-87 - Date & Time Force Cooling Completed: 7-7-87 -	65PM		<b>—</b>			$\sqsubseteq$
		1	Final Temperature After Porce Cooling: 36	<u> </u>		二			二
<u> </u>	(3/1/1)	1/2	Existaining the batch temperature at 37° -			┼	├		+-
Aim		8/	34 Con ode Then adde						$\vdash$
Jun I			Date & Time Addition Started: \$ 11787 5	O PM					
	0.24	10821	Diazolidinyl:Urea:Methyl Paraben:Propyl Paraben:Propylene Clycol (Cormaben II)	960 Ga.	c.198167	HC.	3W	HP	<u>s</u>
		<del> </del> -	Date & Time Addition Completed: 7/7/6/ 5:35	PM		<del> </del>			上
			Rapidly force cool the batch with mixing to	14:5	, 55 1656	. ۋ	Ġı: _		二
		<del>                                     </del>	31° - 33° C.	#000	D158 696		Τά ΗΤ		上
		(0)	Initial Temperature Before Force Cooling: 35 C	010			:	<del></del>	-
		1105	Date & Tinu Force Cooling Started: 7-7-67-	450-1			二		二
		10-1-	Pute & Time Force Cooling Completed: 7-7-87- Final Temperature After Force Cooling: 320	Enors					上
	A fan.	2000	Stop mixing.	<u> </u>		┼—	├	├	╂
							=		$\vdash$
			Heasure actual volume using a calibrated dip stick. 350 L.			#	二		二
-/-	_/	<del> </del>	Adjust the batch to its final volume using the	<b></b>		<del> </del>			
			product's average density (of 1.015 Gm./ml. or			<del> </del>	<del></del>	<u> </u>	$\vdash$
*/			Kg./L.) by adding:			170	10	AR	1/2
	130 to	105310	Purified Water, U.S.P./N.F. q s. to 400 Kg.	44 I		1 ARC	KD-	MI	1 1/2
				(Gal.		1	<u> </u>		$\sqsubseteq$
	(1987)	KO	Mix the batch until uniform maintaining the			#	二		二
n	10/2-10	<b>9</b>	Apperature at 310 - 370 c;	<b> </b>		<u> </u>			上
二 对	1 11 7	/3/8/	Date & Time Batch Uniformity Attained: 7-7-87 400 PM			+	<del> </del>	<del></del>	<del> </del>
=(()	Y	7-/-				1			1
	,	+	Circulate the batch through a Sonolator using a suitable orifice. Operate the sonolator at	<del> </del>		1	<u> </u>	<u> </u>	<del>                                      </del>
	UN'ST		a suitable operating pressure. Dikard The in	that about 5	elleres (19X	\$3/4	The_	057	Ki.
	7/12/11/11		Justed Hornida the Sometato						
7	וודחער לילולי	FF 1114 NO	COCON INITIALING WOOD DONG THEFT AFFIN THEIR FIGURATION	TO FITHER THE BACE (	O THE LACT DAGE				

.ORM 122	14  Rev. 11/86	6)	PILOT BATCH P	NGE 5 of 6	WALGREEN L	ABORA	TORIE	S, INC	<u>.                                    </u>
TITLE	3H INSECT	r/arthroi	POD REPELLENT LOTION W	1	CONTROL NO.		•		
			FORM & MEASURE Cream		(	HB	7		
IODA 1TAG	PTED 6-30	1-87	SUPERSEDES	,			•		
ISSUE(		18	SUPERSEDES DATE: 7/5/	7			-		
1999OVED	BY. Q	To E	1 True DATES 7-6	07	MAT'L O.K.				
			, · ,	•	ALC. REQ. ALC. %				
FREPRODUC	TION CHECKE	D BY:	da DATE: 7-	6-8/	SERIAL NO. CODE NO.				
ORDER NO	)		- AMOUNT FORMULA NO		W.O. NO.				
	<b>≯ ¥/¥</b>	WPN	INGREDIENTS	QUANTITY	INGRED.	MSRE	CKD	ADD	
	<del> </del>		Orinice Sizo: Medium Size Papie (D.	065)	NUMBER	YS	BY	BY	BY
			Sonoistion Uperting Pressure (p.s.1.): 4	M/)				二	丰
		<u> </u>	Sometiment of the state of the			-	-	<del> </del>	┼
			Sonolator Uned: O 2					$\sqsubseteq$	二
	1-		Checked for Clounlineus By fully frante Date & Time: 7-7-87 - 500 (002)			<del>                                     </del>		<del> </del>	+-
		<u></u>	Date & Time Sunclation Started: 7-7-97	6.58 P.M.					$\perp$
		/	Continue circulation until the entire batch	7.20 P.M.					1-
	1111/1/2	<u> </u>	Continue circulation until the entire batch has been sonolated 1 1/2 times.				<b> </b>	<b> </b>	匚
									上
		<del> </del>	Date & Time Circulation Through the Sonolatok Completed:	<b></b>			<del> </del>	├	┼—
(100)	30) [7.1]								二
All 100	1 4/1	<del>                                     </del>	Force cool the batch to 31° - 33° C.  maintaining the temporature no more than 33°			├	<del> </del>	├─	╂─
<u>े रहा</u>	1 3/3/1	7	C.						
( -	11/1/5	<del> </del>	Initial Temperature Before Force				<u> </u>	<b> </b>	$\vdash$
<u>'</u>			Cooling:						$\Box$
		<b> </b>	Date & Time Force Cooling Starter Date & Time Force Cooling Completed:				<del> </del>	<del> </del>	┼─
			Final Temperature After Force Cooling:	1-00					二
	<del> </del>	<del> </del>	Transfer the batch to clean dry pony bowls	* Please )	101	0.5	100	<del> </del>	╂──
71 <del>2 413</del>	1/11		through the Sonolator as before through a 30	A 30 mes	h Stanless	Str	N	a-	二
Klan	(Sentoto	<b></b>	mesh stainless steel screen.	has nu	t available	کیا	D A	2026	₩ <u></u>
9-3-31	10/1/181		Screen Used:	was si	notated a		tille.	In	\$
Ler h			Checked for Cleanliness By: Date & Mice:	bony	how be hit	m	th&n	37	da
A SI	75	Net	M. g. the Brown distanced = 18 kgs.	Serce		1	7	0	
11/12	97	<b></b>	Ono. of Bowls Used: Checked for Cleanliness Bys & Caulius	Januar		10	V <del></del>	/ 7	├
	M'0		Inte & Time: 7-7-37- 2000 pr-1	$\mathcal{O}_{0}$	11-1-2003	47	7/7/	17	
	<del> </del>		Bowl Number Net Weight of Product (Kg.)	<i>∪\K</i>	a (3m)	<u> </u>	<del>                                     </del>	<del> </del>	-
			1 /03.3	<u> </u>	7-8/				二
			2 /n6·/ 3 /03·0		-			<del></del>	┼─-
			4 76:5						$\sqsubseteq$
			Total Wt. 388.9 Kg.						上
-			Sample according to the following instructions						<del> </del>
			and submit the samples to Quality						二
	<del> </del>		Control/Quality Assurance for approval.	·					-
-	<b></b>		500 ml. sample from bottom of kettle 500 ml. sample from top of kettle		<del></del>				厂
	J	<del>}</del>	30 ml. sample from bottom of kettle in sturilo		-				<del> </del>

NOTICE: ANY PERSON INITIALING. WORK DONE, MUST AFFIX THEIR SIGNATURE TO EITHER THIS PAGE OR THE LAST PAGE.

bottle
30 ml. sample from top of kettle in sterile

	4-{Rev 11/86	1	PILOT BATCH PAGE	E 6 01 6	WALGREEN L	ABORA	TORIE!	5, INC	
TITLE	3H INSECT	/ARTHROP	OD REPELLENT LOTION WPW	20186	CONTROL NO.				
C A WITY_	400 Kg. (	394 L.)	FORM & MEASURE Cream		CHE	37		•	
DAT - OPT	ED <u>6-30</u>	-87 /~	SUPERSEDES	Ĺ	·				
ISSULfi		Kest	Hold III . DATE: 7/6/87						
APPROVED I			1- Trus DATE: 7-6-	87	MAT'L O.K. ALC. REQ.				
	ON CHECKE	- 1	da DATE: 7-6-8	-, 87	ALC. % SERIAL NO.				
			AMOUNT FORMULA NO.		CODE NO. W.O. NO.				
	% W/W	WPN	INGREDIENTS	YTITHA!IO	INGRED.				
	·		707	BOTTOM	1	BY	BY	BY	81
			Suppled By: // Kaulin Supple Date & Time: 7-7-87 7-05/11:	17-7-81 72811	<del>-  </del>	<del>  </del>	<b> </b>	<del> </del>	┨—
			Quality Control/Quality Assurance						
			Approval: Date & Time of Quality Control/Quality	<del></del>	<del></del>			<b> </b> -	╀
	1		Assurance Approval:						二
			Theoretical Weight: 38/ 400 Kg.				<u> </u>	<u> </u>	├
	3311		Actual Weight: 388.9 189						
	1//07	281-	Tereent Actual Tield: //	7/1	-				├
	VIII		Calculated By: // DA Can L	<i>ii</i> ·					二
<del></del>			Checked by:			1			一
			Chilary ha						
		<del> </del>	SW = Ster Wayleako				_		<del> </del>
-!			BNW = Contest. a Litertland (Bh.Ca)	2	· ·	$\Box$	=	$\dashv$	
$\exists (\exists$							$\equiv \downarrow$		
			AR = Aubuf Eawin					$\Box$	
			60 100 4 5						
			CO CANAMAN,						
			MC- MilA Rank	<del></del>					
			141 = 1 Jan 1 Range						
		<del></del>				<del>  </del>			<del> </del>
							=		
						<del>  </del>		-	
							=		_
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						$\square$			$\vdash$
			<u> </u>						
							=	=	
			<del> </del>						
							二	=	_
-			<del></del>		1				
		<del></del> ;						二	
								<del></del>	
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	HOTICE	: ANY PE	RSON INITIALING WORK DONE, MUST AFFIX THEIR SIGNATURE TO I	EITHER THIS PAGE OR	THE LAST PAGE.		•		-

# Walgreens

#### CERTIFICATE OF ANALYSIS

3M INSECT/ANTHROPOD REPELLANT LOTION WPN: 20186 #CHB7
Date of Manufacture 7-7-87

TEST	SPECIFICATIONS	TEST RESULTS
APPEARANCE	White, viscous cream with slight DEET odor.	Smooth, white lotion.
DENSITY	0.995 - 1.035 g/ml	1.004 @ 25°C
VISCOSITY*	150,000 - 250,000 cps (#TE @ 0.6 rpm)	21,600 cps (#TB @ 5 rpm) @ 25°C
рН	6.9 - 7.5	7.4
m.DEET	31.58 - 36.75%	Top = 35.3% w/w Bottom = 34.0% w/w
Bacti: TPC	Not more than 100/ml	10

^{.*}VISCOSITY: Conducted on a RVT Model Brookfield Viscometer

^{*}Using #TE @ 0.5 rpm the reading on the scale is 2.0 Using #TE @ 1 rpm the reading on the scale is 2.2 Using #TE @ 5 rpm the reading on the scale is 5

^{*3}M Lab Sample #462-15: Viscosity = 35,600 cps (#TB @ 5 rpm)

Bases on Pilot Batch CHB7

101M 122	4-(Rev. 1]/86	1	PRODUCTION TRIAL		WALGREEN LA	MORATO	RIES, IN	ic.
4	jh insec	r/arthroi	OD REPELLENT LOTION WP		ONTROL NO.		•	
BUAN	2900 Kg.	(3253 L.	FORM & MEASURE Lotion		C.	PP7		
· • • • • • • • • • • • • • • • • • • •			, SUPERSEDES	,, L			<del></del>	
gSUED-BY.			STAR MILE DATE: 7/20/	<i>\$7</i>				
PPROVED	er, Sh	illi		87	MAT'L O.K ALC. REQ			
EPRODUC	TION CHECKE	D BY.	Butty Chrene DATE, 7-2		SERIAL NO.			
			AMOUNTFORMULA NO		CODE NO	·		
	*			11	INGRED.	10800	1	1
;	\$ ¥/¥	WPN	INGREDIENTS	QUANTITY	CONTACL	l sx l		
	<del>\</del>		Date & Time Batch Started: ( . LPAN /2/8	Palentalia by	A Strait			
		,	PART 1: Place in a 600 gallon stosm jacketed kettle equipped with a sweep action mixer:	Call thicked by	7-13-1			
	1		Kettle Used: WOO. GAL GROEN	FACTOR: 0.	90625			
	<del> </del>		Mixer Used: SWEEP Checked for Cleanliness By: Appli	REASON: Shot	THE BANK	HERMA	- AM	- 130%
	-		Date & Time: 6. HAM / 1818	$\rho$	Tracel			<b></b> .
		2	Date & Time Added: 6:33AM 7.27.87	. (	The same of the sa			
100	35:95	10531C		1042.6 2350-4 Kg.		A	20	
1	<b>\</b>		Add with slow mixing, to the water:	(1043 (1150 L.) (27/ 1304 Gal.)	3.	1	4 6	-00
3	<b>]</b>		7.77	FOAM		2 5		<del>  </del>
	1.64	10386	/ Amorphous Silicon Dioxide (Cab-0-Sil M-5)	47.6 52.5 Kg.	1780767	1	70 6	1/2
Train	2.04	10,00	Date & Time Addition Completed: 727877.					100
	<del></del>		Slowly start to mix.					
			Wash the walls of the kettle with a small amount of Purified Water, U.S.P./N.F. (WPN		<del> </del>		<del></del>	<del></del>
			10531C).					
			Increase the speed of the propeller mixer so that the batch is mixing thoroughly. CAUGH 10	WORM TO THIS INS		12787	(3)	
			Add while mixing:	KETTLE HAS ONLY OU	STEED /S	47.	43	
			Date & Time Addition Started: 7-27-17	115 AT 7:48AM				
	2.26	11318	/Polyethylene Glycol (7) Glyceryl Ether	65.5 72.3 Kg.	1866767	25	III R	A
			(Liponic EC-7)					
	0.98	10384	Polyethylene Glycol (8) (Carbowax 400)	98.5 32-4 Kg.	C185767	15		142
	0.7	12929	Magnesium Aluminum Silicate (Veegum)	20.3 22.4 Kg.	V339727	129	1/1	THE
	<u> </u>		Date & Time Addition Completed: 72787	8.;SAM				
			Sample as per the current Ointmont Sampling Procedure.					丰富
			Submit 4 fl. oz. sample to 3M Personnel.					
			Sampled By: Asket					1-1
			Sample Date & Time: 7.27.87 (109 Am)					<del> </del>
			Now heat Part I to 480 500 C (8/-8 5				1	+=1
			maintaining the temperature no higher than 500 Q. while mixing.				二二	1
							二二	<b>#</b>
					13	ZC		1
	NOTICE	: ANY PE	RSON INITIALING WORK DONE, MUST AFFIX THEIR SIGNATURE T	O EITHER THIS PAGE OR TI	E LAST PAGE.			: 7

Based on Pilot Batch CHB7

ORP 1224	(Rev. 11/86		PRODUCTION TRIAL		WALGREEN L		LTORIE	S, INC	-
STLE	In Insec	T/ARTHROS	POD REPELLENT LOTION WI		CONTROL NO.			~_	-
UAN (	1200 Kg.	(325)	form & MEASURE Lotion	,		~ <b>7</b>			
;	· •	9	SUPERSEDES		CP	P			
ELLED AV.	, t	18	CHARAMA DATE 7/20	167					
SOLO PII	· A		ain II. Myl- D'ATE: 7-22		MAT'L O.K.	·-·			<del></del>
FROVED	مريدان	444	The same of the sa	,	ALC. %	<del></del>			·
1		,			SERIAL NO CODE NO				
RDER NO		· · · · · · · · · · · · · · · · · · ·	AMOUNT FORMULA NO		WO NO .			<u>`</u>	
	<b>'</b> ≯ ¥/¥ '	WPN	INGREDIENTS	QUANTITY	INGRED. CONTROL NUMBER	MSRD BY	CKD	1	ŧ .
				7.87		二	二	二	二
			Pinal Temperature Attained: 36°C	7.87 Vd 7-27)	7	$\leftarrow$	├	┼─	┼──
, ,		7.	71-83	NAR 7-27-87					$\downarrow =$
.——-	<u> </u>	<b> </b> -	Maintain Part I at 482 500 €. with constant mixing until ready to be added to	Q		┨──	┼	┼─	<del> </del>
			Part II.				1		1
·			PART II: Place in a 1000 gallon steam jacketed			┼─	┼──	├─	┼
			kettle equipped with sweep action mixing, side				1	1	1
	,		acrapers and agitator:		<del> </del>	<del> </del>	<del> </del>	╂	<del> </del>
			Kettle Used: /WGA GREO				二	1	1
	<u>_</u>	ļ	Date & Time: 12787 975 AM			┼	<del> </del>	<del> </del>	+
ユコ			Date & Time Addition Started: 9:20914 75	7.87			二		1
	26.916	12098	/85:7.5:7.5 mole ratio lso-Octyl Acrylate:Stearyl Methacrylate:Acrylic Acid Terpolymer-21.66% in Deet	780.6 861.3 Kg.	C86476)	25	MA		HATE
	77.077	10574		7.27 445 5-14	0.799787	MA	NAV	1	1/5
	13.914	10554	/ N.N-Diethyl-M-Tolumnide (MGK Diethyltolumnide 95% Neta Isomer Minimum)	403.6 443.7 KR.	12/11/67	<del>177</del>	THE STATE OF	1//	100/12
			Date & Time Addition Completed: 12 200M	72787	7,2	<u> </u>		<del> </del>	<u> </u>
			Begin to mix Part II with aveep action mixing				1		
		<del> </del>	and side scrapers at a slow speed.	<u> </u>		<del> </del>	├─	<del> </del>	┼
			Sweep Action Mixer Speed Setting: 3						
		ļ	Heat Part II to 590 - 610 C. maintaining		<del> </del>			-	
			the temperature no higher than 61° C. while		<del></del>		<b></b> -	<u> </u>	ļ
			mixing.						二
			Date & Time Heating Started: / OPM 7-77  Date & Time Heating Completed: 7-20-17-/:10/m	87	<del> </del>	<del> </del>			
			Final Temperature Attained: 6/6	1 / -	Apr	26	77		
	,	ļ	Add carefully while mixing: Sollowing	2DA-	9 - 6-61	ب	12	1	
			1 Hm	NACZ	-27-87 34	-27	Da.		
			Date & Time Addition Started: 7-7757/.5D		NYE 2	100	272		
	1.65	12931	/ Polyethylene Glycol (82) Glyceryl Monotallowate (Varonic L148)	47.9 -52.8 Kg.	C795967	1/3	Sil	AX	18
		10010		10° 0 00 6° V	X-10/-2/-	A	2777	10	-
	0.65	12930	/ Polyethylene Clycol (200) Clyceryl Monotallowate (Varonic L1420)	18.9 20.6 Kg.	C796767	دم	ş W	NA.	16
	1.55	11313	1	86.0 40-EV-	0784767	15	خ	Post	1
_ <b>(</b> †	1.75	11317	/ Clyceryl Stearate/Sodium Lauryl Sulfate (Lexemul AS)	45.0 19.8 Kg.	C/01.0/	440	1	200	acc
	24)	11315	/Propylene Glycol Dicaprylate/Dicaprate (Lexol	19.9 27.1 Kg.	0783167	K	SW.	mod	in
	2.41	11717	PG865)	47·7 451 NS.	103/0/	22		434	The same
<del></del> -	0,65	13000	Cetyl Palmitate (Waxenol 816)	18.9 20.0 Kg.	C783707	PC	يو ک	1	
					12-1-1	ښو			
<u>-</u>	NOTICE	: ANY PE	I RSON INITIALING WORK DONE. MUST AFEIX THE'R SIGNATURE T	O-EITHER THIS PAGE OR T	HE LAST PARE		<b></b>	بـــا	

Based on Pilot Batch CHB7

110RM 1224	4 (Rev. 11/86	3)	PRODUCTION TRIAL	AGE, 3 H 6	WALĞREEN L	ABORA	TORIE	S, INC	<b>:</b> .
	<del></del>		POD REPELLENT LOTION WI		CONTROL NO.				1
	3900	, 3857	.) FORM & MEASURE Lotion	7					
`	<b>A</b>	^			C	PP7			
ATE ADOPT	7-2:	2-87	SUPERSEDES	17	·				
SSUED BY.			DATE: 7/22/	8/					
PPROVED	17. Sh	ila	en XX Ary DATE 1-25-		MAT'L O.K. ALC. REQ.				-
EBA O DATE			da i DATEI 7		ALC. %				
				•	SERIAL NO.				
PRDER HO.			AMOUNT FORMULA NO.		W.O. NO.				
	≴ ¥/¥	WPN	INGREDIENTS	QUANTITY	INGRED, CONTROL NUMBER	1 8%	BYO	BY,	, 8Y
	2.2	10026	Polypropylene Clycol (15) Stearyl Ether 39	STORE DA KE.	18/11/67		36		
			(Arlamol E)			<del> </del>	1		
	2.2	10046	/Cetyl Stearyl Alcohol (Adol 63)	13.8 20.4 Kg.	C797767	15	7	A L	177
			Date & Time Addition Completed: 7-27-87-2:10	in		二			二
		<del> </del>	Heat Part II to 810 - 830 C. along with	<u> </u>	_	<del> </del>		<del> </del>	<del> </del>
			moderate agitation maintaining the temperature no higher than 83° C						
				<u> </u>					
		-X	Sample as per the current Ointment Sampling Procedure.						-
		1-7				1			二
		<del>   </del>	Submit 4 fl. or. sample to 3M Personnel.						}
=(	D. 1-		Sampled By: Amily Handles Sample Date & Time: 7-27-8/						
	17/27/87		2.03.03.09						
100	27-22-13	<del>                                     </del>	Sweep Action Mixer Speed Setting: 4-3 Date & Time Heating Started: 22 82 -1014						<del> </del>
			Date & Time Heating Completed: 7-27-87-3:05's Pinal Temperature Attained: 82°C	m .				*2,	
			Date & Time Part II Completely Dissolved &						
			Uniform: 7-27-17 3-05-P-M.		<u>,,,</u>				
			Add slowly to Part II while mixing:				二		
			Date & Time Addition Started: 7-27-87-3:107-						
	0.5	11616	Cellulose, 2-Hydroxy Ethyl Ether (Natrasol 250 HR)	14.5 14.0 Kg.	C182161	B	70	Y / 2	Pla
			Date & Time Addition Completed: 7-27-87-3:20	74			-	#	
			Mix Part II until a good dispersion is						
			obtained, about 15 minutes.			二			
			Mixing Time: 3:20pm to 3:35pm			$\dashv$			
	,		Date & Time Part II is Uniformly and Well			$\Box$	二	$\Box$	
			Dispersed: 7-17-17 3.35 p.M.			二.	十	_	
			Increase Part II mixing to give rapid agitation.			$\dashv$	<b>—</b>	二	
						二	二		
			Sveep Action Mixer Speed Setting: 4	US 7-27-87	<del></del>				
			With Part II at BPO - B30 C. alorly add	NAC 7-770		<b>二</b>	<b>=</b>	二	
一世 十			Part I.			士	士	士	
			Reheat Part II if necessary to 81° = 83° C.			<del></del>	<b>-</b>	一	
						寸	丰	二	<del>~</del>
					<del>                                     </del>			<b>一</b>	<i>[</i>
	NOTICE	ANV	AND MALLING MADA DONG THE TANK		1000			<i>_</i> .	
<b>-</b> · ·	MUTICE:	ANT PER	SON INITIALING WORK DONE, MUST AFFIX THEIR SIGNATURE TO	) EITHER THIS PAGE OR T	HE LAST PAGE		•	6/	<i>*</i>

Pilot Batch CHB7

ORM 122	4 [Rev. 11/8	6)	PRODUCTION TRIAL -	AGE 4 of 6	WALGREEN I			ES, IN	c.
. <u> </u>	3K THER	T/ARTHRO	OPOD REPELLENT LOTION W	20186	CONTROL NO.	•			
	3900	3857	.) FORM & MEASURE Lotion			- 07	,		
•	•				C	pp7			
ATE ADOP	TED	22-87	SUPERSEDES	11	L				
SUED BY			Brind Al Alling DATE, 7/20	1/87					
SPPROVED	7	. 7.		8-87	MAT'L O.K. ALC. REQ.				
					ALC. 76				
EPRODUCT	ION CHECK	ED BY	da DATE: 7	-23-8/	SERIAL NO.				
RDER HO.			AMOUNT FORMULA NO.		W.O. NO.				
		1	Ţ	T	INGRED.	7	T	T	T
	% W/W	WPH	INGREDIENTS	YTITHAUQ	CONTROL				
·							芷	兰	土
<del></del>		<del> </del>	Initial Temperature: 82°C  Date & Time Reheating if Necessary					<del> </del>	-
:		<del></del>	Started: NOT NECESCARY	<del></del>			<del> </del>	╁~~	+-
			Date & Time Reheating if Necessary			1			1
			Completed: NoTNECESCA) +						L
		<del> </del>	Pinal Temperature Attained: 82 C	<del>- </del>			┼	<del> </del>	╂
		<del> </del>	Date & Time Part I Addition Started: 7-2>-	1 - 2:30PM		<del> </del>	┼──	╂	╁—
		1	Date & Time Part I Addition Completed: 2-27	-87-3:55 Pm		+	<del>                                     </del>	+	+-
		╀	Rinse Part I kettle with a small amount (about			┦	ļ	<b>├</b> ──	╀—
		<del> </del>	5 gallons) of Purified Water, U.S.P./N.P. (WP. 10531C) and add the rinsings to the batch	^}		<del></del>	<del> </del>	┼	╂
		<del>                                     </del>	(combined Parts I & II).	<del> </del>		+	┼──	<del> </del>	+-
				<u> </u>		1	<b>†</b>		1_
<u> </u>		ļ	Initial Temperature: 50°C						_
( 4		<del> </del>	Reheat combined Parts of I & II to 810 -			┼	<del> </del>	┼	╂—
-		<del> </del>	83° C.		<del>.  </del>	<del> </del>	<del> </del>	<del> </del>	┼─
						1	<del>]                                    </del>	1	1
		ļ	Date & Time Reheating of Necessary						
		<b> </b>	Started: 7.25.87-3:55Pm			<b>↓</b> -	├	<del> </del>	╀—
		<del> </del> -	Date & Time Reheating if Necessary  Completed: 5-25-87-4:///m	<del> </del>		<del> </del>	<del> </del>	┼	╂──
			Pinal Temperature Attained: 82°C	1	<del></del>	<del>                                     </del>		1	1
				-x recirculate o	small auround				
		ļ	Mix the batch for 20 minutes at 81° - 83°	I of the property	th ivnixe	1	↓	<del> </del>	<del> </del>
		<del> </del>	_ C.	complete unitary	with at the p	\$ <b>X</b> 0_	├	├	┼─
		<del> </del>	Mixing Time: 4:1570 to 4:357M	9/	& Westerko	7/28	767	<del> </del>	<del>                                     </del>
				Ġ	esteuren	1-28	E		
		<u> </u>	Force cool the batch with mixing to 37° -	<u> </u>	<del></del>	<del> </del>	<u> </u> -	<b> </b>	<del> </del> —
		<del> </del>	, , , , , , , , , , , , , , , , , , ,	<del> </del>		1	ļ	<b> </b>	<del> </del>
			Adjust the mixing speed while force cooling to		/	2.0	D- k 9	* GF_	
		ļ	maintain an adequate mixing action. Avoid the		16:32	1.8	D-kg	111	<b> </b>
		<del> </del>	entrapment of air.	<del> </del>	#20001E 010821	6.0	u kg		<del> </del>
			Sweep Action Mixer Speed Setting: 4	i .		, ,,,,,,			
			ANTOIOR SPEED -9		16:38				<b> </b>
			Initial Temperature Before Porce		#100008		ग्रम्		-
<del></del>		<b></b>	Date & Time Force Cooling Started: 9-27-87	N: Yahm	010821/	بعب	7. 9	1	<del> </del>
			Date & Time Porce Cooling Completed: 1.22.97	-8:20m		1	-	ايبام	
	<u> </u>		Pinal Temperature After Porce Cooling: 3/9						
			Then add:						<b> </b> -
			Iden acc:	<u> </u>				<b></b>	
			Date & Time Addition Started: 7-27-87-8:25	(PM				二	
				<b>)</b>	17.00=1	<i>M</i> 4	27,	10	B
<b>-(1</b> +	0.24	10821	/ Diagolidinyl:Urea:Hethyl Paraben:Propyl L' Paraben:Propylene Glycol (Germaben II)	6.160 2.650 Xz.	C283717	334 B	hmi	474	ALL
		<del></del>	terenantionitana Afacol (Asimposu 11)		1				<u> </u>
			Date & Time Addition Completed:7-27-87-8:10	rM				$\Box$	
	—— <u> </u>				-				
		<del></del>	· · · · · · · · · · · · · · · · · · ·	<del></del>	<del> </del>	<b></b> ┼			
					7.7				
•	NOTICE	: ANY PE	RSON INITIALING WORK DONE, MUST AFFIX THEIR SIGNATURE T	O EITHER THIS PAGE OF T	HE LAST PAGE.				

ORM 122	4 jaov. 11/86	)	PRODUCTION TRIAL	GE 5 •1 6	WALGREEN L			s, inc	
ITLE	SH THERE	/ARTHROPO	OD REPELLENT LOTION WA	N 20186	CONTROL NO.				
_	290	3857				0.01	o <b>7</b>		
	, 3200 Kg.					CPI	71		
ITE ADOP	TED <u>7-22</u>	-87	SUPERSEDES	1/0					
SUED BY			PARAL MIL. : DATE: 7/94/	8)	r				
, SPROVED	BY. Sh				MAT'L O.K. ALC. REQ.				
,					ALC. %				
PRODUCT	ION CHECKE	D BY	da U DATE:	-237/	SERIAL NO.				
ROER NO.	·		AMOUNT FORMULA NO		W.O. NO.				
	\$ ¥/¥	WPN	INGREDIENTS	PTITHAUQ	INGRED. CONTROL NUMBER	MSRD BY		ADD BY	CKD BY
		/	Aeasure actual volume using a calibrated dip			=			
	<b></b>		stick.						
		}	Actual Volume of the Batch: 2700 L.			<del> </del>			
;			Adjust the batch to its final volume using the Product's average density (of 1.015 Gm./ml. or		}	1			<b> </b>
			Kg./L.) by adding:						
	0.0.0 to	105310	Purified Water, U.S.P./N.F. q.s. to 3200 Kg.	/57 L.		100	NAR		WAL
	100		(3153 L.) (2857L.)	( Kg.)			7.1.	7	
	<del></del>	<del> </del>		( Gal.)	<u>'</u>	-			
			Rapidly force cool the batch with mixing to						
			) · · · · · · · · · · · · · · · · · · ·						
_ , _ ;		-*	Initial Temperature Before Force Cooling: 3/4		, .	<del> </del>			
720	ME		Date & Time Porce Cooling Started: 8/17	VEFDED:					
Kari-	7/2/-50		Pinal Temperature After Force Cooling: NAT	MILLICABLE OF	51 Var 197				
77714	73~		Stop mixing.	1,1111111111111111111111111111111111111					
			Date & Time Batch Uniformity Attained: 7-27-85-9:00pm						
			Circulate the batch through a Sonolator using						
			medium size (0.065) orifice and a 30 mesh						
			stainless steel acreen attached to the end of the Sonolator. Operate the sonolator at a		<del></del>	<del>  </del>			
			350-450 p.s.i. pressure. Discard the initial						
	far.	1	about 5 gallons (19 kg.) of the product passed through the Sonolator and through the screen.					-	
	1/2/7/18	2)	The Actual martity of the Bookent Dissance?	2/K55.					
	Man pap	10	Orifice Size Used: : 065 Screen Used: Nove						
			Actual Sonolation Operating Pressure 400 PLT (p.s.i.) Employed: 400 PLT						
							=	$\Box$	
	<b></b>		Sonolator Used: 707-0, May 15 Checked for Cleanliness By: How Hitherell	<u> </u>					
			Date & Time: 7-27-87-9:0577				$\Box$		
			Date & Time Sonolation Started: 7-27-87-10-75	im _					
			Date & Time Sonolation Completed: 2-23-23-10	YUPM			-		
			Continue circulation until the entire batch				二	二	
			has been sonolated 1 1/2 times. "					-	
			Date & Time Circulation Through the				=	二	
<u>`</u> -			Sopolator Completed:						
			BOWN 1-3 SOUNTION STAPTED 10:30AM				-	$\dashv$	
								二	
<b>,</b>		ļ	BOWS \$3 CODONATION STATION P. A. ASPA SOUCHTIN FINISARD 18,33P		A.F.S'OF	-		_	7
	HOTICE	: ANY PE	RSON INITIALING WORK DONE, MUST AFFIX THEIR SIGNATURE T						*

# OVEXLENT & P (591)

BLADE ON SOMOLATOR ABAD MOVED CLOSER TO ORIFICE SWOMATION PRESSURE KEPT AT 800P. S.I. AST 1-20.87 BOWE GANS ? - STATED-DOTTING DELET- FROM FINED DATE TIME J. 21-87-4:10/4

CHASED ORIFHE THOM DES TO . DET -SSCHWARDEN PREMINE HEAT FOR 800 P.S.I. Bows 8- 3-28-87-4:40PM ... 9 - 6:45A7-28-67 ...10 - 7:00th ... 76 03VOM. 11 - 7:10PM PARQUEODN CH 12-930 pm __ ,OH \$30; 13-940 AM 14 - 7:15M - 7-2987 16 - T:20AM 7:25AM 17- 10:0044 18 - 11:15A4 11:25AM 746 PM 23- 810 PM 24- 930 pm

THE SH INSECT/ARTHROPOD REPELLENT LOTION WPN 20186  PAUA. (346) KR. (3173 L.)  SUPERSEDES  SUPERSEDES  SUPERSEDES  PROVED BY	PORM 1224 R	ter. 11/86)		PRODUCTION TRIAL	AGE 6 01 6	WALGREEN L	ABORA.	TORIE	S:iNC	· ·
DUAL (1855).)  FORM & MASSURE LOSION  STREAM OFFICE AND SUPERSTORES  SINCE BY. SUPERSTORES  STREAM OF THE PRIOR OF THE BUILD TO DATE. TO D	וווגב או	INSECT	/ARTHROP	OD REPELLENT LOTION W	£ .	CONTROL NO.				
STREADONED BY.  DATE.  DATE.  DATE.  PRODUCTION CHECKED BY.  ACC. 840.  PROPER NO.  AMCUNI  FORMULA NO.  DATE.  PROPER NO.  AMCUNI  FORMULA NO.  FORMULA NO.  DATE.  PROPER NO.  AMCUNI  FORMULA NO.  FORMULA NO.  DATE.  PROPER COOL DEC SECRET TO 310 - 390 C.  STRIAL STREAM STR	DUA. ( 32	7900 180 Kr.	3857	) FORM & MEASURE Totion			0.5	ה <b>י</b>		
PRODUCTION CHECKED BY.  AMOUNT TOEMULA NO.  PORCE NO.  AMOUNT NORREDENTS  OUANITY  POPER COOL THE BAICH TO 31V - 33V U.  BAILE RESONANCE ON THE BAICH TO 31V - 33V U.  BAILTIRING THE TEACH TO STORE COULDE STATE 33V U.  BAILTIRING THE TEACH TO STORE COULDE STATE 33V U.  DATE A TIME PROPER COULDE STATE	ATE ADORSED	A 2 22	97 1	land warmen			UY	'		
PRODUCTION CHECKED BY.  AMOUNT TOEMULA NO.  PORCE NO.  AMOUNT NORREDENTS  OUANITY  POPER COOL THE BAICH TO 31V - 33V U.  BAILE RESONANCE ON THE BAICH TO 31V - 33V U.  BAILTIRING THE TEACH TO STORE COULDE STATE 33V U.  BAILTIRING THE TEACH TO STORE COULDE STATE 33V U.  DATE A TIME PROPER COULDE STATE	A POOPLED	1-22	~/(	bil. AD AM	//s		<del></del>			
PRODUCTION CHECKED BY.  AMOUNT TOEMULA NO.  PROPER NO.  AMOUNT INCREDIENTS  ORDER NO.  AMOUNT INCREDIENTS  ORDER NO.  POPER COOL THE BAICH TO 31V - 33V U.  BAILTAINING THE BAICH TO 31V - 33V U.  BAILTAINING THE TEACH TO SET UND STORE UND STORE UND SET U.  INCREDIENT STORE COOLING STARTES THE STORE COOLING STARTES THE	STUED BY	7	-14	DATE: HODE	8/	MAT'L O.K.			_	
SERIAL NO. CODE NO.  AMOUNT FORMULA NO.  S V/V WPN INGREDIENTS  OUANTITY CONTROL ASSO CED ADD C NUMBER SY IT BY IT BY IT SY IT	PPROVED BY	16	<u>vill</u>	Carno XI. O lange DATE 7-2-2	-87	ALC. KEQ.				
SERVE NO. AMOUNT FORMULA NO. WO. NO.    SERVE COOL THE DETCH TO 310 - 350 U.   INGRED. CONTROL MASE   SY   SY   SY   SY   SY   SY   SY	EPRODUCTION	CHECKE	3Y1	da U DATEI 7	-23-87	SERIAL NO.				
WPN   INGREDIENTS   QUANTITY   ASSO CED	DRDER NO			AMOUNT FORMULA NO	·	CODE NO.				
Relatating the temperature no more than 330   U. U.		\$ ¥/¥	WPN	INGREDIENTS	QUANTITY	CONTROL	, ,			CKD BY
Relatating the temperature no more than 330   U. U.				Force cool the batch to 31° - 33° U.	<u> </u>					
Initial Temperature Before Yorce  Cooling: 3/C  Bate & Time Force Cooling Started: AT NET DED  Pate & Time Force Cooling Started: AT NET DED  Pate & Time Force Cooling Started: AT NET DED  Pate & Time Force Cooling Started: AT NET DED  Pate & Time Force Cooling Started: AT NET DED  Sample according to the following instructions and substit the samples to Quality  Control/Quality Assurance for approval.  500 ml. sample from bottom of kettle  500 ml. sample from bottom of kettle  30 ml. sample from bottom of kettle  30 ml. sample from top of kettle  30 ml. sample from top of kettle  30 ml. sample from top of kettle in startle  bottle  Sampled By:  Sampled By:  Sample Date & Time: ATT NEW Made on the formula and			<u> </u>	maintaining the temperature no more than 330						<del> </del>
Cooling: 3/4.  Date a Time Force Cooling Started: AM NF. DED  Date a Time Force Cooling Completed: MIT MANIALE PROPERTY OF THE	<del></del>			C		1		=		
Date & Time Force Cooling Started: ANT WILLIAMS  Date & Time Force Cooling Completed MIT WILLIAMS  Final Temperature After Force Cooling: Not MANIABLE  Sample according to the following instructions  and submit the samples to Quality  Control/Quality Assurance for approval.  500 ml. sample from bottom of kettle  500 ml. sample from top of kettle  30 ml. sample from top of kettle in sterile  bottle  Sample Br:  Sample Br:  Sample Br:  Sample Br:  Sample Brie & Time: MITATEM MANIA WAN MANIABLE  Date & Time of Wality Control/Quality  Masurance  Approval: Canglandan  Date & Time of Wality Control/Quality  M. Assurance Approval: 7-ANN (100 MITATEM)  Acceptable Yield Range: 98 - 1025  Percent Actual Yield:  Calculated By:  Checked By:  Sample Maniable Mitatematics  Maniable Mi				Initial Temperature Before Force	<del> </del>	<del></del>	<del>  </del>			
Date & Time Force Cooling Completed: MIT WILLIAMS  Final Temperature After Force Cooling: AND MANUALIST  Sample according to the following instructions and submit the samples to Quality  Control/Quality Assurance for approval.  500 ml. sample from bottom of kettle  500 ml. sample from bottom of kettle  30 ml. sample from bottom of kettle  30 ml. sample from top of kettle in sterile  bottle  Sample Br:  Sample Br:  Sample Br:  Milly Theoretical Workshop of Cooling of Manualist Control/Quality Assurance  Approval: Toolyoutland on the Manualist Control Quality  Assurance Approval: Toolyoutland on the Manualist Control Quality Control/Quality  Theoretical Veight:  Accuptable Tield Range: 98 - 1025  Percent Actual Veight:  Calculated By:  Checked By:				Cooling: 3/C.	//= = X = X					
Sample according to the following instructions and subsit the samples to Quality Control/Quality Assurance for approval.  500 ml. sample from bottom of kettle 500 ml. sample from top of kettle 30 ml. sample from bottom of kettle in sterile bottle 30 ml. sample from top of kettle in sterile bottle  Sample Br:  Sample Br:  Sample Br:  Sample Br:  Matt thanks under many of the time of Quality Assurance Approval:  Approval:  Assurance Approval:  Theoretical Veight:  Acceptable Yield Range:  Percent Actual Yeight:  Acceptable Yield Range:  Percent Actual Yeight:  Calculated By:  Checked By:  Checked By:  Checked By:				Date & Time Force Cooling Started: ANT Date & Time Force Cooling Completed: WIT	WELDED 1	1/1/1	<del>                                     </del>			
Sample according to the following instructions and subsit the samples to Quality Control/Quality Assurance for approval.  500 ml. sample from bottom of kettle 500 ml. sample from top of kettle 30 ml. sample from bottom of kettle in sterile bottle 30 ml. sample from top of kettle in sterile bottle  Sample Br:  Sample Br:  Sample Br:  Sample Br:  Matt thanks under many of the time of Quality Assurance Approval:  Approval:  Assurance Approval:  Theoretical Veight:  Acceptable Yield Range:  Percent Actual Yeight:  Acceptable Yield Range:  Percent Actual Yeight:  Calculated By:  Checked By:  Checked By:  Checked By:				Finel Temperature After Force Cooling: Not	APPLICABLE DE	7/7/8/81				
and subsit the samples to Quality  Control/Quality Assurance for approval.  500 ml. sample from bottom of kettle  500 ml. sample from top of kettle  30 ml. sample from bottom of kettle in sterile  bottle  30 ml. sample from top of kettle in sterile  bottle  Sample Brice Affine:  Sample Brice Affine:  Coulity Control/Quality Assurance  Approval:  Approval:  Approval:  Assurance Approval:  Approval:  Assurance Approval:  Assura	<del></del>				<u> </u>	1-11-7-7		$\dashv$		
Control/Quality Assurance for approval.  500 ml. sample from bottom of kettle 500 ml. sample from top of kettle 30 ml. sample from bottom of kettle in sterile bottle 500 ml. sample from top of kettle in sterile bottle  Sample By:  Sample By:  Sample Bate & Time: //// // // // // // // // // // // //				and submit the samples to Quality						
500 ml. sample from top of kettle  30 ml. sample from bottom of kettle in sterile bottle  30 ml. sample from top of kettle in sterile bottle  Sample Brice from top of kettle in sterile bottle  Sample Date & Time: ////////////////////////////////////				Control/Quality Assurance for approval.				=	$\Box$	
500 ml. sample from top of kettle  30 ml. sample from bottom of kettle in sterile bottie  30 ml. sample from top of kettle in sterile bottle  Sampled By:  Sample Date a Time: ////////////////////////////////////				500 ml. sample from bottom of kettle		-				
bottle  30 ml. sample from top of kettle in sterile  bottle  Sample By:  Sample Date & Time: ATTANAM  Quality Control/Quality Assurance  Approval: Carlandan  Date & Time of Quality Control/Quality  Assurance Approval: 7-AS.VN \ 100 VM  Actual Veight:  Acceptable Yield Range: 98 - 1025  Percent Actual Yield:  Calculated By:  Checked By:  Checked By:				500 ml. sample from top of kettle				二		
Sample from top of kettle in sterile  bottle  Sample By:  Sample Date & Time: 2317 A. From  Quality Control/Quality Assurance  Approval: Anglowidge  Date & Time of Quality Control/Quality  Assurance Approval: 7-AS Y. 100 Pm  Acceptable Yield Range: 98 - 1025  Percent Actual Yield:  Calculated By:  Checked By:  Sw - Muldbuydonto  Acceptable Yield Range: 98 - 1025	- ( +-						$\neg$	$\Box$	$\Box$	
Sample By:  Sample Date & Time: / STY / Provided And the formula were made approval: Assurance Approval: 1-28.57					P. Note:	<del></del>			$\dashv$	
Sample By:  Sample Date & Time: 7877/27897  Quality Control/Quality Assurance  Approval: Contact on the Approval: Control/Quality  Dete & Time of Quality Control/Quality  Assurance Approval: 1-28.97 (100 PM 3M Approval)  Theoretical Veight: 288/3189 Kg.  Actual Veight: Acceptable Yield Range: 98 - 1028  Percent Actual Yield: Calculated By:  Checked By:  SW = Manufacturance  Acquality Control/Quality  Acceptable Yield Range: 98 - 1028  Calculated By:  Checked By:  Manufacturance  Manufactur					i		E		刀	E
Sample Date & Time: 787 A. 1997  Quality Control/Quality Assurance Approval: 200 Audion Date & Time of Quality Control/Quality Assurance Approval: 7-AV.YO (100 PM 3M MATTER)  Theoretical Veight: 388 318; Kg. Actual Veight: Acceptable Yield Range: 98 - 1025 Percent Actual Yield: Calculated By: Checked By:  SW = Matthewards  Matth				Sampled By:	MIN TAW	yes and	//Y 4/	7764D	514	44
Quality Control/Quality Assurance  Approval: Asplanta Min and authory of the Assurance Approval: 1-18.87 (100 Pm 3M history 100 Pm 3M hist				Sample Date & Time: 72887 12.7599	made in the	in som	rla	wen	20	MA
Dete & Time of Quality Control/Quality  Assurance Approval: 7-NS-VO V-00 VR 3M MARKET  Theoretical Weight: 388/ 318; Kg.  Actual Weight: Acceptable Yield Range: 98 - 1025  Percent Actual Yield: Calculated By: Checked By:  Checked By:  Theoretical Weight: 388/ 318; Kg.  Actual Veight: 388/ 318; Kg.  Actual				Quality Control/Quality Assurance	10/1-0/10	1 .0 . 1	- [	4		
Theoretical Weight: 388 318; Kg.  Actual Weight: 788 318; Kg.  Acceptable Yield Range: 98 - 102;  Percent Actual Yield:  Calculated By:  Checked By:  Sw = Swalandad Arab (188)  Meant to mat substitute to the control of the control				Date & Time of Quality Control/Quality		1 . 0 1	- /\ \/	9 1	~7	
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Acceptable Yield Range; 98 - 1029  Percent Actual Yield:  Calculated By:  Checked By:  S.W Menchanto						1-6-4	graff	40	44	
Fercent Actual Yield:  Calculated By:  Checked By:  Sw = Struckburgerko  Allow Arter hims 13:22  Meanul & Internal Strucks  Meanu				Actual Veight:				76X	8/	
Calculated By:  Checked By:  Sw = Stewhbuylonko  She alone was not slove  Keawe & Intel was 1221						1 July	ta.	-4		
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# OVEXLEAF 8/69%)

After sonolating at pressure of 370, 500, 600, 700 \$ 800, it was decided the the 800 poi gave the best vesults. The oily oppositive scemed to be move finely dispersed at this pursure.

The final ~ 34 of the batch will be sonolated at 800 psi.

Store Washerth

As the product is being sonderled into pany bowb, it should be mixed at a low spread to provent seperation.

# Walgreens

#### CERTIFICATE OF ANALYSIS

3M INSECT/ARTHROPOD REPELLENT LOTION 20186 #CPP7 7-27-87 Date of Manufacture

TEST	SPECIFICATIONS	TEST RESULTS
APPÉARANCE	White, viscous cream with slight DEET odor.	White lotion, as if separated into tiny droplets.
DENSITY	0.995 - 1.035 g/ml	1.006
VISCOSITY*	150,000 - 250,000 cps (#TE @ 0.6 rpm)	32,900 cps*
рН	6.9 - 7.5	7.5
m.DEET	31.58 - 36.75%	35.3% w/w

Bill Lange

Asst. Mgr. QC/QA

^{*}viscosity; Conducted on a RVT Model Brookfield Viscometer *Using  $\#TB\ @\ 5$  rpm

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			- C	PP7	
		DIY 2 av. or. FORM & HEASURE . Creek			:
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ened by —		DATE 7/1/87			•
PROVED BY		ary- ame My Kulse DATE 7/2/87	12 THUCHA	209	
PRODUCTION	CERTO	BY Michael C. Son DATE 7-28-87	N9 10. 347	<u> </u>	· 
	VPK.	PACKAGING COMPONENTS	TTTTTAUP	OFFERIO BY	CEECE
	1	(R(12(f))			
	<del> </del>	Fackaging Line Checked for Cleanliness By:  Date & first Flory (100ff)	rate Bur (1/4)	<del> </del>	
		7/ 25/27	1. 7.26 - 37		
<del></del>	<del> </del>	All latel and packaging sections of the line have been checked for residual packaging. All such packaging material has been removed.	THE FLE BY 8.57		
	1	The state of the s	2-27-57		
		Checked By: ( )C-	Totali		
	<del> </del>	/ Date & Time: #/20/17 /- 1-00/19   \$\forall P(\forall \gamma')			
		Quality Control/Quality Assurance Approval to Run: 17 A Kul			
	<del> </del>	Date & Time: 7/28/87 1:15/A			
		Date & Time Packaging This Control Started: 7/1/17 1:/5	Vivu		
	<b> </b>	101100			
		Line Used: 15	<del> </del>		
		Filling Heads Tube Coder Carton Coder			
<u>.</u>	<del> </del>	Set By:			<u> </u>
		Date: 1/08/87 7/08/87	-		
	<del> </del>	Tixe: 11/00 AM 1:00 fm		1.	<del></del>
		Place 2 av. oz. only of:			
	20186	3M Insect/Arthropod Repellant	12), -3200 KG		
			2881		
		Into:			
	61069	2 or. HDPE Tubes (1 1/2" x 3 1/2") with turnet dispensing closure	-56, 338-	100	NZW
		22/400 finish clive drab color, one color printing.	50721		
		Suitably crimp the tube closed, imprinting the Control Number on the	46.469		<del></del>
		crimp.		-	
		Place the tube into the following shipping carton (46 per minuming			
	1	carton): Which is stenciled			
4/24)	140845	Partition 3m Insect Arthograph Kerell			
2. Kilor		Lotion (PPM)	at 1,173		
	10846	Shipping Carton	x alpho 1.177	11.32	~/x~
7. Lune		Now apply the following label to each filled shipping carton	704	11.27	YC.,
	1/22200				
d 34	1 12301	5 x 2 1/2 Pressure Sensitive Stock, Permanent Adhesive Shipping Label, White with Black Print	1.175		_
ske		Now print Gontrol Number on shipper label,			
		Attach the following example of labeling used to package this control	1		
		Shippor label (or stenciling if used) showing control number.			
		Collect all unused labeling material.			سبب
		Collected By: 7. Color Date & Time: 7-30-77 1:30 PM			<u></u>
		Collect all unused packaging material.			
	но	TICE! ANY PERSON MILITALING WORK DO.	ACE	<del></del>	

JI PAGE

MASTER PACKAGING FORMULA באר וניו שני. 11/\$6; VALOREZA LABORATORIUS. INC. 2 of CONTROL NO. 3X INSECT/ABTHEOPOD REFELLANT LOTION, 2 AV. OZ. PPOF 028021 UYT: Crean . 56,338 Only 2 av. os. PORM & MEASURE 6-30-87 (0:1g.) Supersedes ORDER NO. SUED BY £1,501 THUCHA PROVED BY PRODUCTION CERCITED IN OFFERED CERCICO אקע CHARTITY BI H PACKAGINO COMPONENTS Inspect the line to insure removal of the above, unless the packagin materials are to be used for the next control. Inspected By: 8.00 B. P. Date & Time: 7-31-77 Date & Time Peckaging This Control Finished: 7-3e-17_ NOTICE. ANY PERSON NITIALING WORK DONE MILLY AFFIX THEIR SIGNATURE TO THE LAST PAGE

THE RESERVE OF THE PROPERTY OF

# TIV-PHOUESS CUNIRULS

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ITAAUS	Tr _ 56,338 0=1	g 2 av. oz.	FORM & NEASUR	E Cresz			CPP?
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			me Th. Fubre DAT	E		_ THOURY	
REPROD	UCTION CHECKED	EY E.char	10 r : DAT	T-28-87	<u> </u>	KS. NO	3475
				Calc. Ey	Checked Ev		
	Stated Fil		2 84. 02.	. i/A	K/A ·		
	7111 Used		62		10, 1, 21		
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	REIKU	t mange: 1	rom total weight to no more tha				14. K Libe 71
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	7/25/57	2,20%	+1297	MA. Enlo	OR	O.F	makey
	7-28-87	4:38	+1500	3.6.	O.K.	0.K	).c.
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	7-29-87	25:11	+ 2gm	913 carron	7 4	or_	1913 tekan
المراجعة	anged	TEFE C	en from white	to Ale	30 (See)		
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		يرولي =	troloted of 800 can	1 Pec .051	-5 NAR 25)-U		
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	7-28-87	7:07	+ 1 drie	D.C.	D.K.		· · · · · · · · · · · · · · · · · · ·
	7-28-87	7:29	+10th +25m	HH:LL D.C.	0. K.	G/L- O.K.	Dic.
	7-28-87	9:01	7/2m	D.C.	0.K.	U.K.	D.C.
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	J-39-87	1:32	+19m	RT RT	1 3		82
	7-29-87	4:11 PH	1 gm +10gm	To colon	Q.K.	, <b>4</b> / <b>0</b> / <b>C</b>	Del
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7-30-87 3								
D2021128 D2202121								
PRODUCT RECONCILIATION	Cas	100	<del></del>	Units	<del></del>	Calo. By	Checked by	·
Snipping Carton Counts		649	42834	·		19 3-90	Untin	
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LABELING RECONCILIATION			<del></del>	<del></del>			<del>``</del>	
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No. of Units Packaged:	42,539		7.55	Unitar		-	·	
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SAMPLING INFORMATION	·		·				^,	
Take 5 completely packaged unitate the end of the run.	ts from the	packaging l	ine. 4 at th	e beginning o	f the rup	and I		
43 CHE WIND DI 100 TUD.							1 /4% ·	
		Semples	1 - 4	Sagnle 5	:			
Sampled By: Sample Date & Time:		7/25/19	1120 P.M	7/30/57	7320AF			
•				11.25/17				
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R. R Janlowsh

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P8.8

GG ONLY 028021 2 FL.OX.

3M INSECT/ARTHROPOD REPELLANT LOTION CPP7

MANUFACTURING PROCESS: 1000 Gallon Batch of Insect/Arthropod Repellent

CKD By

MSRD

BY

CKD

BX

ADD

BY

PROCESS	QUANTITY	INGRED. CONTROL NUMBER ^
PART I:	<del></del>	
Use a 600 gallon steam jacketed kettle equipped with a sweep action mixer.		•
Kettle Used: Mixer Used: Checked for Cleanliness By: Date/Time:	- - -	
Date/Time Batch Started:	_	
Add to the Kettle:		-
Purified Water, U.S.P./N.F. Date and Time Added:	3000 lbs. (360 gals.)	,
Add with intermittent mixing to the water:		
Amorphous Silicon Dioxide (Cab-O-Sil M-S)	136.9 lbs.	
Date/Time Addition Started:	- -	
Wash the walls of the kettle with a small amount of Purified Water, U.S.P./N.F.		
Add the following while mixing:		
Date/Time Addition Started:	-	
Polyethylene Glycol (7) Glyceryl Ether (Liponic EG-7)	188.6 lbs.	
Polyethylene Glycol (8) (Carbowax 400)	81.8 lbs.	
Magnesium Aluminum Silicate (Veegum)	58.4 1bs	
Date/Time Addition Completed:	<del></del>	

(

INGRED. **PROCESS** QUANTITY **MSRD** CKD ADD CONTROL BY NUMBER BY BY Take a 4 ounce retain sample of Part I. Sampled By: Sample Date/Time: Now heat Part I to 81-83°C (177-182°F) maintaining the temperature no higher than 83°C while mixing. Date/Time Heating Started: Date/Time Heating Completed: Final Temperature Attained: Determine the volume of Part I at 81-83°C Volume Determined: Keep Part I suitably covered and maintain at 81-83°C with constant mixing until ready to add to Part II. Check the volume of Part I immediately before the addition of Part I to Part II and compensate for any loss of volume due to evaporation by the addition of Purified Water U.S.P./N.F. as required. PART II: Use a 1000 gallon steam jacketed kettle equipped with sweep action and agitator mixing. Kettle Used: Checked for Cleanliness By: Date/Time Add to the Kettle: IP-III Polymer 25% in DEET 1946.9 lbs. (3M, 41-4202-1922-6) Date/Time Addition Started: N_nN-Diethyl m-Toluamide (MGK Diethyltoluamide 95% 1460.4 lbs. meta isomer minimum) Date/Time Addition Completed:

CKD

BY

PROCESS	QUAN	TITY.	INGRED. CONTROL NUMBER
Begin to mix Part II with side wall scrapers and agitator at a slow speed.	<u></u>		
Sweep Action Mixer Speed Setting: 3 Agitator Speed Setting: 5			
Start heating Part II to 81-83°C (177-182°F taking care the temperature rises no higher than 83°C while mixing.	)		
Date/Time Heating Started:			
Add carefully while heating to 81-83°C the following with continued mixing:			
Date/Time Addition Started:			
Polyethylene Glycol (82) Glyceryl Monotallowate (Varonic LI48)	137.7	lbs.	
Polyethylene Glycol (200) Glyceryl Monotallowate (Varonic LI420)	54.2	lbs.	
Glyceryl Stearate/Sodium Lauryl Sulfate (Lexemul AS)	129.3	lbs.	
Propylene Glycol Dicaprylate/ Dicaprate (Lexol PG 865)	201.1	lbs.	
Cetyl Palmitate (Waxenol 816)	54.2	lbs.	
Polypropylene Glycol (15) Stearyl Ether (Arlamol E)	183.6	lbs.	
Cetyl Stearyl Alcohol (Adol 63)	183.6	lbs.	
Date/Time Addition Completed:			
Continue to heat Part II to 81-83°C with moderate agitation. Maintain 81-83°C until all the ingredients are fully dissolved.			
Date/Time 81-83° Attained: Date/Time Part II Completely Dissolved and Uniform:			

ADD By

CKD By

MSRD

BY

CKD BY

•		
PROCESS	QUANTITY	INGRED. CONTROL NUMBER
Take a 4 ounce retain sample of Part II.		-,
Sampled By: Sample Date/Time:		
Add slowly to Part II with moderate mixing:	•	
Cellulose, 2-Hydroxethyl Ether (Natrosol 250 HR)	41.7 lbs.	
Date/Time Addition Started: Date/Time Addition Completed:		
Mix Part II until a good dispersion is obtained - about 15 minutes.		•
Mixing Time:to		
Check the volume of Part I and adjust to originally measured volume with Purified Water, U.S.P./N.F. if required.		
Date/Time Checked: Volume of Phase I: Final Adjusted Volume of Phase I:		
Adjust temperature of Phase I to 81-83°C.	•	
Initial Temperature: Date/Time of Reheating:		
Started: Completed: Final Temperature Attained:		
Increase Part II mixing to give rapid agitation. Use higher speed settings for the sweep action mixer and the agitator if required.		
Sweep Action Mixer Speed Setting: 4 Agitator Speed Setting: 9		
With Part I and Part II both at 81-83°C slowly add Part I through a pump and hose to Part II. Maintain vigorous mixing of the batch using the sweep action mixer and the agitator.		
Date/Time Part I Addition Started: Date/Time Part I Addition Completed:	····	

CKD By

MSRD BY ADD By CKD By

Rinse Part I kettle with a small amount (about 5 gallons) of Purified Water, U.S.P./N.F. and add the rinsings to the combined Part I and Part II batch.  Reheat combined Parts I and II to 81-83°C.  Initial Temperature: Date/Time Reheating Started: Date/Time Reheating Completed: Final Temperature Attained:  Mix the batch for 20 minutes at 81-83°C.  Mixing Time	PROCESS	QUANTITY	INGRED. CONTROL NUMBER	MSRD BY	CKD BY	ADD BY	C B
Initial Temperature: Date/Time Reheating Started: Date/Time Reheating Completed: Final Temperature Attained:  Mix the batch for 20 minutes at 81-83°C.  Mixing Time	(about 5 gallons) of Purified Water, U.S.P./N.F. and add the rinsings to the						
Date/Time Reheating Started: Date/Time Reheating Completed: Final Temperature Attained:  Mix the batch for 20 minutes at 81-83°C.  Mixing Time	Reheat combined Parts I and II to 81-83°C.						
During the mixing time clear 10 gallons of the product from the valve at the bottom of the 1000 gallon kettle and add the cleared material back to the batch. Repeat this clearing procedure at 70°C, at 60°C and at 50°C.  Date/Time Material Cleared: Cleared By: Date/Time Cleared Material Added to Batch:  Force cool the batch to 37-39°C (98-102°F) with mixing.  Adjust the mixing speed while force cooling to maintain an adequate mixing action and yet avoiding air entrapment.  Sweep Action Mixer Speed Setting: 4 Agitator Speed Setting: 9  Initial Temperature Before Force Cooling: Date/Time Force Cooling Started: Date/Time Force Cooling Completed: Final Temperature After Force Cooling:	Date/Time Reheating Started: Date/Time Reheating Completed:						
During the mixing time clear 10 gallons of the product from the valve at the bottom of the 1000 gallon kettle and add the cleared material back to the batch. Repeat this clearing procedure at 70°C, at 60°C and at 50°C.  Date/Time Material Cleared: Cleared By: Date/Time Cleared Material Added to Batch:  Force cool the batch to 37-39°C (98-102°F) with mixing.  Adjust the mixing speed while force cooling to maintain an adequate mixing action and yet avoiding air entrapment.  Sweep Action Mixer Speed Setting: 4 Agitator Speed Setting: 9  Initial Temperature Before Force Cooling: Date/Time Force Cooling Started: Date/Time Force Cooling Completed: Final Temperature After Force Cooling: Then Add:	Mix the batch for 20 minutes at 81-83°C.						
the product from the valve at the bottom of the 1000 gallon kettle and add the cleared material back to the batch. Repeat this clearing procedure at 70°C, at 60°C and at 50°C.  Date/Time Material Cleared: Cleared By: Date/Time Cleared Material Added to Batch:  Force cool the batch to 37-39°C (98-102°F) with mixing.  Adjust the mixing speed while force cooling to maintain an adequate mixing action and yet avoiding air entrapment.  Sweep Action Mixer Speed Setting:  Agitator Speed Setting:  9  Initial Temperature Before Force Cooling: Date/Time Force Cooling Completed: Final Temperature After Force Cooling: Then Add:	Mixing Timeto						
Date/Time Material Cleared: Cleared By: Date/Time Cleared Material Added to Batch:  Force cool the batch to 37-39°C (98-102°F) with mixing.  Adjust the mixing speed while force cooling to maintain an adequate mixing action and yet avoiding air entrapment.  Sweep Action Mixer Speed Setting: Agitator Speed Setting: Initial Temperature Before Force Cooling: Date/Time Force Cooling Started: Date/Time Force Cooling Completed: Final Temperature After Force Cooling: Then Add:	the product from the valve at the bottom of the 1000 gallon kettle and add the cleared material back to the batch. Repeat this clearing procedure at 70°C, at 60°C and						
Adjust the mixing speed while force cooling to maintain an adequate mixing action and yet avoiding air entrapment.  Sweep Action Mixer Speed Setting:4 Agitator Speed Setting:9  Initial Temperature Before Force Cooling: Date/Time Force Cooling Started: Date/Time Force Cooling Completed: Final Temperature After Force Cooling: Then Add:	Cleared By:		°C 70°C	60	°C	50°C	
to maintain an adequate mixing action and yet avoiding air entrapment.  Sweep Action Mixer Speed Setting: 4 Agitator Speed Setting: 9  Initial Temperature Before Force Cooling: Date/Time Force Cooling Started: Date/Time Force Cooling Completed: Final Temperature After Force Cooling: Then Add:							
Agitator Speed Setting: 9  Initial Temperature Before Force Cooling: Date/Time Force Cooling Started: Date/Time Force Cooling Completed: Final Temperature After Force Cooling: Then Add:	to maintain an adequate mixing action and						
Date/Time Force Cooling Started:  Date/Time Force Cooling Completed:  Final Temperature After Force Cooling:  Then Add:	Sweep Action Mixer Speed Setting: 4 Agitator Speed Setting: 9						
	Date/Time Force Cooling Started:		-				
	Then Add:						
Diazolidinyl:Urea:Methyl Paraben:Propyl Paraben:Propylene Glycol (Germaben II) 20.0 lbs.  Date/Time Addition Completed:	,	20.0 lbs.					

C

PROCESS	QUANTITY
Rapidly force cool the batch to 31-33°C (88-92°F) with mixing.	L
Initial Temperature Before Force Cooling: Date/Time Force Cooling Started: Date/Time Force Cooling Completed: Final Temperature After Force Cooling:	
Measure batch volume (using a calibrated dip stick).	
Actual Volume of Batch: gallons.	,
Adjust the batch to its final volume by adding:	
Purified Water, U.S.P./N.F. q.s. to 1000 Gallons.	
Mix an additional 30 minutes to a uniform batch.	
Mixing Time From: to Date/Time Batch Uniformity Attained:	
Circulate the batch through a Sonolator (or other suitable homogenizer) discharging the product below the surface of product through the port at the top and back of the 1000 gallon kettle. Operate the sonolator using a small (0.051-S) orifice at 775-825 p.s.i. pressure with the knife edge of the sonolator adjusted as close to the orifice as possible Discard the initial product (about 5 gallons 19 Kg) passed through the sonolator.	J or
Sonolator Used: Checked for Cleanliness By: Date/Time:	
Orifice Size Used: Sonolator Operating Pressure Used: Actual Quantity of Product Discarded:	
Date/Time Sonolation and Circulation Started:	

INGRED. CONTROL NUMBER

MSRD BY CKD By ADD By CKD By

Continue sonolation and circulation as above until the entire batch has been sonolated 1-1/2 times.

Date/Time Circulation through the Sonolator Completed:	
Force cool the batch to 31-33°C if needed.	
Initial Temperature Before Force Cooling: Date/Time Force Cooling Started:	
Date/Time Force Cooling Completed: Final Temperature After Force Cooling:	
- Tillar remperature Arter rolle cooring.	

Sample the final lotion according to the following instructions and submit the samples to Quality Control/Quality Assurance for approval.

500 ml sample from bottom of kettle 500 ml sample from top of kettle 30 ml sample from bottom of kettle in sterile bottle 30 ml sample from top of kettle in sterile bottle

Sampled By:
Sample Date/Time:
Quality Control/Quality
Assurance Approval:
Date/Time of Quality Control/Quality
Assurance Approval:

Theoretical Yield:

995 Gallons

8428 Lbs.

3823 Kgs.

5.3 Appendix C

# PACKAGE SPECIFICATIONS FOR TUBE, CAP, SHIPPING CARTON AND LABEL:

TUBE

Style: Plastic Tube

Material: High Density Polyethylene

Color: Custom color olive drab

Size: 2 ounce, nominal

Dimensions: 1-1/2" Diameter x 3-3/16" Long (Fill Length)

Orifice size: 0.187; Head: 22/400

Weight: 7.71g + 0.16g

Printing: 2 color - White and Black with

TP-46 epoxy resin barrier coating

Source: Tubed Products Inc.

Easthampton, MA

Copy:



6840-00-XXX-XXXX
INSECT/ARTHROPOD REPELLENT LOTTON

TYPE (XXX)
Federal Specification XXXXXXX
Contents: 2 Fluid Ounces

Repels mosquitoes, biting files, chiggers, deer files, fless and stable files. Also repels terrestrial leeches in tropical areas where pest occurs.

Provides 95% or greator protection against mosquitoes for 12 or more hours under normal use conditions.

ACTIVE INGREDIENTS: N,N-Diethyl-m-toluamide 31.58%; Other isomera 1.75%; Inert ingredients 66.75%.

FOR EXTERNAL USE ONLY Keep out of reach of children.

Caution – Avoid contact with eyes and lips.
In case of eye contact, flush with plenty of water.
Do not apply to excessively sunburned or damaged skin.

Contract No. DAMD17-85-C-5017

DIRECTIONS FOR USE

It is a violector of Federal lev to use this product in a manner inconsistent with its histelling.

BOW EPPLICATION

Squeeze into one hand 2.5 ml of repetent, a strip squel in length and with to the olegans on the state of the tube. But hands to the olegans on the state of the tube. But hands together and apply thoroughly in a term layer to both forearms. He additional beton for upon arms. Repeat for other exposed areas, it spert to beat, squeeze lotton from point of hand and street on those since and each. Avail Contact With Eyes seet Lips. Repet at necessary. When hands after application, they powerse certain synthetic technics, plactics, panied or variathed surfaces, Jeond amening on plastic syspiles in fames, popples, welch crystale, etc. WILL HUT DAMARE rylons, cotton or wool fabros. Dispessib Do not stude erroply container. Within container and put in triefs.

Strip Diagram

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Personal Care Profesta/365, 3M Coreer St. Paul, Minnesotz 55144-1009; EPA Reg. No. XX EPA Est. No. XXX CAP

Style: FT

Material: Low Density Polyethylene

Color: Custom color olive drab

Finish: 22/400

Seal Type: Land

Orifice Size: 3.5 mm x 7.5 nm

Part Number: PS 118

Source: Polytop Corporation

Slatersville, RI

#### SHIPPING CARTON

Style: Assembled Partition

Material: 125 lb test, KRAFT Board Type, C Flute

#### Dimensions:

1. One set of assembled partitions consisting of:

Part "A" Size: 12-1/4 x 4-11/16" (7 required)
Part "B" Size: 16-3/8 x 4-11/16" (5 required)

- 2. Assemble Part "A" with Part "B" to form a 48 cell partition
- 3. After assembly knock down flat
- 4. See below for dimensions of Part "A" and Part "B"

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Front Label

Repels mosquitoes, biting flies, chiggers, deer flies, fleas and stable flies. Also repels terrestrial leeches in tropical areas where pest occurs.

Provides 95% or greater protection against mosquitoes for 12 or more hours under normal use conditions.

ACTIVE INGREDIENTS: N, N-Diethyl m-toluamide 31.58% Other isomers 1.75%; inert ingredients 66.75%.

FOR EXTERNAL USE ONLY Keep out of reach of children.

Caution - Avoid contact with eyes and lips. In case of eye contact, flush with plenty of water. Do not apply to excessively sunburned or damaged skin.

Contract No. DAMD17-85-C-5017

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Back Label

#### **DIRECTIONS FOR USE**

It is a violation of Federal Law to use this product in a manner inconsistent with its labeling. SKIN APPLICATION

Squeeze into one hand 2.5 ml of repellent, a strip equal in length and width to the diagram on the side of the tube. Rub hands together and apply thoroughly in a thin layer to both forearms. Use additional lotion for upper arms. Repeat for other exposed areas. To apply to face, squeeze lotion into palm of hand and spread on face and neck. Avoid Contact With Eyes and Lips. Repeat as necessary. Wipe hands after application.

May Damage certain synthetic fabrics, plastics, painted or varnished surfaces. Avoid smearing on plastic eyeglass frames, goggle, watch crystals, etc. WILL NOT DAMAGE nylon, cotton or wool fabrics.

Disposal: Do not reuse empty container. Wrap container and put in trash.

Personal Care Products/3M 3M Center St. Paul, Minnesota 55144-1000

EPA Reg. No. XX EPA Est. No. XXXXX

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BAT	CH SIZE: 1000 GALLONS	-BATCH SIZE: BOO GALLONS	BATCH SIZE: 686 GALLONS				
	NOV 187 ₂	NOV 187	NOV 187 -				
	OPTION 1	OPŢION 2	OPTION 3				
LABOR HOURS 168	\$4,651	\$4,651 168	\$4,651 1 <del>82</del> ~				
OVERHEAD	4,382	4,382	4,382				
NATERIALS/SUPPLIES	8	~ <b>8</b>					
TRAVEL	2, 199	2, 199	2,199				
SUBCONTRACTOR & RAW MATERIALS	S: 28, <b>845</b>	23, 428	18,812				
SUBTOTAL	<b>\$</b> 39 <b>,</b> 197	\$34 <b>,</b> 598	<b>\$29,964</b>				
6#A 22.55% IRED 5.38%	8, 843 1, 958	7,861 1,729	6,76 <b>8</b> 1,498				
TOTAL	\$50,900	\$44,111	\$38,222				
FEE 0.86%	<b>\$</b> 8	- \$8	\$8				
TOTAL	\$56,888	\$44,111	\$38,222				
FACITITIES COST OF CAPITAL	\$38	\$38	\$38				
TOTAL PROPOSAL	\$50, 0.30	\$44,141	\$38,252				

NOTE: Possible rounding differences

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Insect Repellant LS9061-0680-USA67

TRAVEL

HHHH MITPC		MONTH	LOCATIO		PURPOSE					MEALS ********	GROUND TRANSP		TOTAL PER TRIP	TOTAL WITHOUT INFLATION ####################################	TOTAL INCLUDIN INFLATIC FIFTH
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